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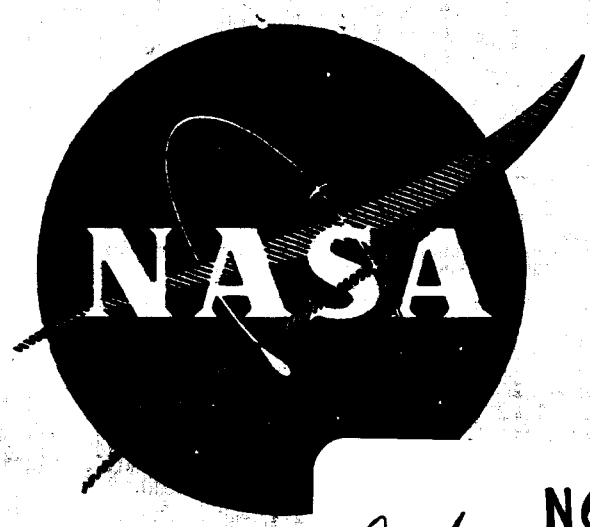

DEVELOPMENT OF DISPERSION STRENGTHENED TANTALUM BASE ALLOY

Third Quarterly Report

by
by R. W. Buckman, Jr. and R. T. Begley

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
UNDER CONTRACT NAS3-2542

Astronuclear Laboratory
Westinghouse Electric Corporation

Astronuclear

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THIRD QUARTERLY PROGRESS REPORT

Covering the Period

December 20, 1963 - March 19, 1964

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2542

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FOREWORD

This report was prepared by the Astronuclear Laboratory of the Westinghouse Electric Corporation under Contract NAS 3-2542. This work is administered under the direction of the Nuclear Power Technology Branch of the National Aeronautics and Space Administration with Mr. P. E. Moorhead acting as Technical Manager.

This work is being administered at the Astronuclear Laboratory by R. T. Begley, with R. W. Buckman serving as principal investigator. Other Westinghouse Astronuclear Laboratory personnel contributing are D. Stoner, R. L. Ammon, G. G. Lessmann, and J. L. Godshall. This report covers the work performed during the period December 20, 1963 to March 19, 1964.

Astronuclear

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I. INTRODUCTION

This is the third quarterly report under Contract NAS 3-2542 covering activities from December 20, 1963 to March 19, 1964. The objective of this program is the development of a dispersion strengthened tantalum base alloy for use in the 2400 to 3000°F temperature range of advanced space power systems. The alloys are being developed primarily from the Ta-W-Hf-C system with limited substitution and/or additions of Re and Mo, Zr, and N for the tungsten, hafnium, and carbon respectively.

A total of twenty-nine 800 gram non-consumably melted compositions at the tantalum rich end of the Ta-W-Hf-C system have been prepared and processed to sheet. Substitutional alloy additions ranged from 6 a/o W + Hf to 16 a/o W + Hf with carbon additions in the range of 0.26 to 3.0 a/o (0.02 to 0.2 w/o).

Fabricability and weldability were affected by the total solute addition, total carbon addition, and the ratio of the reactive (Hf) metal addition to the carbon addition. The carbon addition was effective in promoting dispersed phase hardening at elevated temperatures since strength increases observed in short time tensile properties and increased resistance to creep deformation could only be attributed to dispersed phase strengthening. The creep tests were conducted in ultra-high vacuum (10^{-8} Torr). Paucity of published creep data for tantalum base alloys tested under similar conditions permits only limited comparison of properties.

A series of compositions with substitution and/or additions of molybdenum and rhenium, zirconium, and nitrogen for the tungsten, hafnium, and carbon respectively were melted, upset forged, and processed to 0.04 inch thick sheet. The substitutional solute additions were restricted to a total of 9 a/o. Hot hardness measurements and recrystallization studies show an increase in recrystallization temperature by as much as 400°C and improvements in hot hardness by as much as 10 to 20% by minor substitutions of rhenium, molybdenum, and zirconium for the tungsten and hafnium.

Six compositions melted as two-inch diameter ingots were processed to 0.04 inch thick sheet. Ingot breakdown was accomplished by upset forging or extrusion to sheet bar using a Model 1220C Dynapak unit. Recrystallization behavior, metallography, and room temperature mechanical properties were determined. The addition of 500 ppm carbon to T-111 (Ta-8W-2Hf) and compositions containing a total of 12 w/o W + Hf results in a pronounced increase in the TIG weld bend transition temperature.

II. PROGRAM STATUS

A. MECHANICAL PROPERTY EVALUATION EQUIPMENT

Mechanical properties are being evaluated using room and elevated temperature hardness measurements, short time tensile tests, bend ductility determination, and creep tests. Hardness and short time tensile property measurements are being utilized to define effects resulting from gross compositional variations, and for following metallurgical changes resulting from the various thermal-mechanical treatments. Compositions exhibiting severe degradation of bend ductility as a result of welding are not being considered for detailed mechanical property evaluation. The primary criterion for strength evaluation is the resistance to creep deformation. One hundred hour creep tests are being used to select compositions exhibiting the best creep characteristics.

1. Hot Hardness

Hardness measurements at elevated temperatures are made at pressures below 5×10^{-5} Torr in the apparatus shown in Figure 1. The operation of the hot hardness equipment is described by Begley, et al.[1] Sheet specimens, 0.040 inch thick, are riveted to a molybdenum mount using unalloyed tantalum or T-111 wire. The test surface is metallographically prepared prior to making the hardness determination.

2. Tensile Tests

Tensile testing is carried out according to the procedures recommended by the MAB. Room temperature tests are conducted at a strain rate of 0.005 in/in per minute through the 0.6% offset yield strength and at 0.05 in/in per minute until fracture occurs. A constant strain rate of 0.05 in/in per minute is used for the elevated temperature tests. A detailed outline of specifications for tensile testing including test specimen configurations is listed in Appendix I.

3. Creep Testing

The systems used for creep testing are shown in Figure 2 and embody essentially the same features as described by Hall[2] except that the internal load capacity is greater. Major components of the system are the vacuum system, furnace and power supply, the loading train, and the optical strain measuring device.

The vacuum system consists of a stainless steel bell jar chamber, a feed through sump, the weight chamber, and a 400 l/s sputter ion pump. Cryogenic sorption pumps, shown in Figure 3, are used to reduce the system pressure from

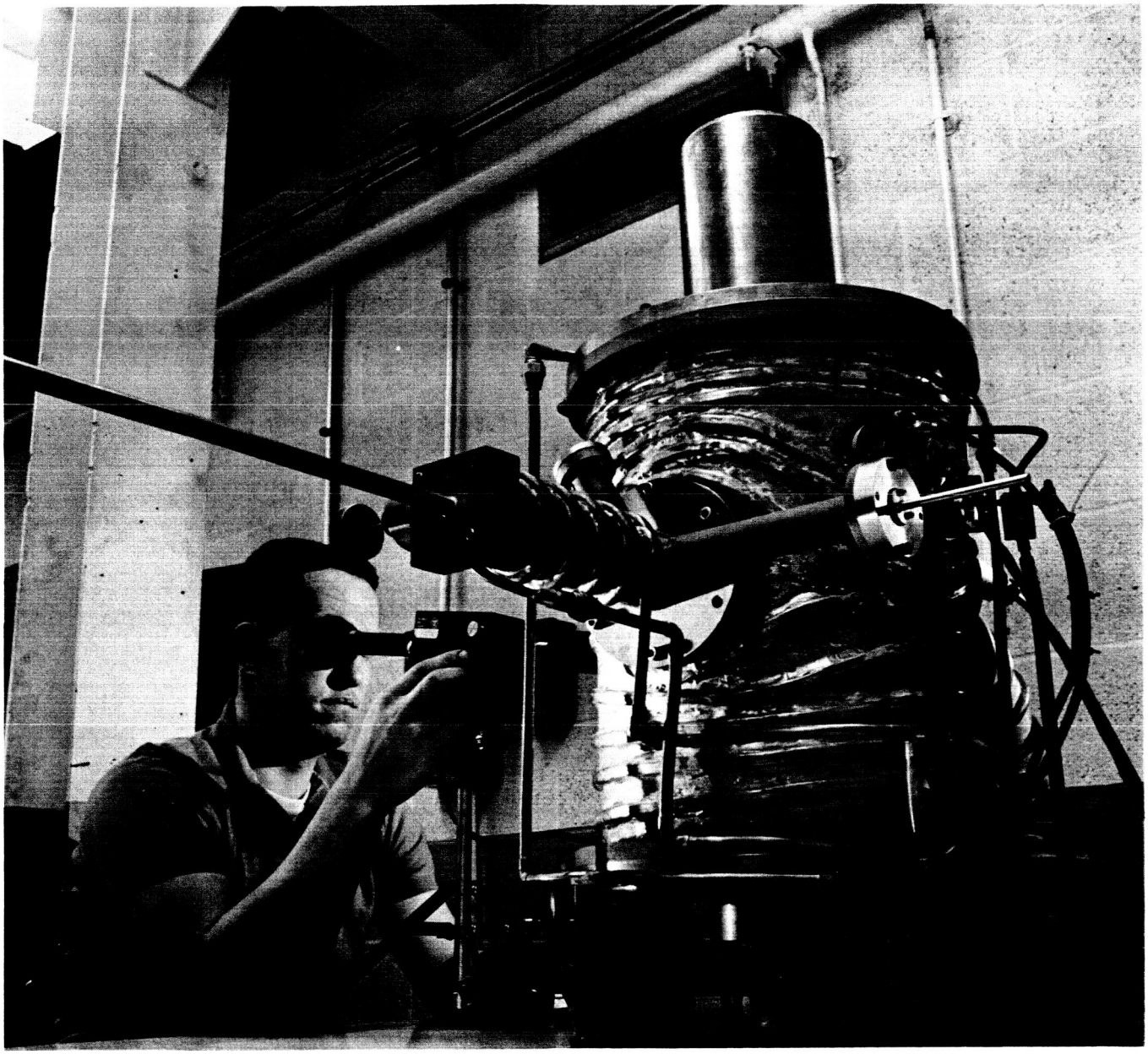


FIGURE 1 - Hot Hardness Test Equipment

FIGURE 2 - Ultra-High Vacuum Creep Laboratory

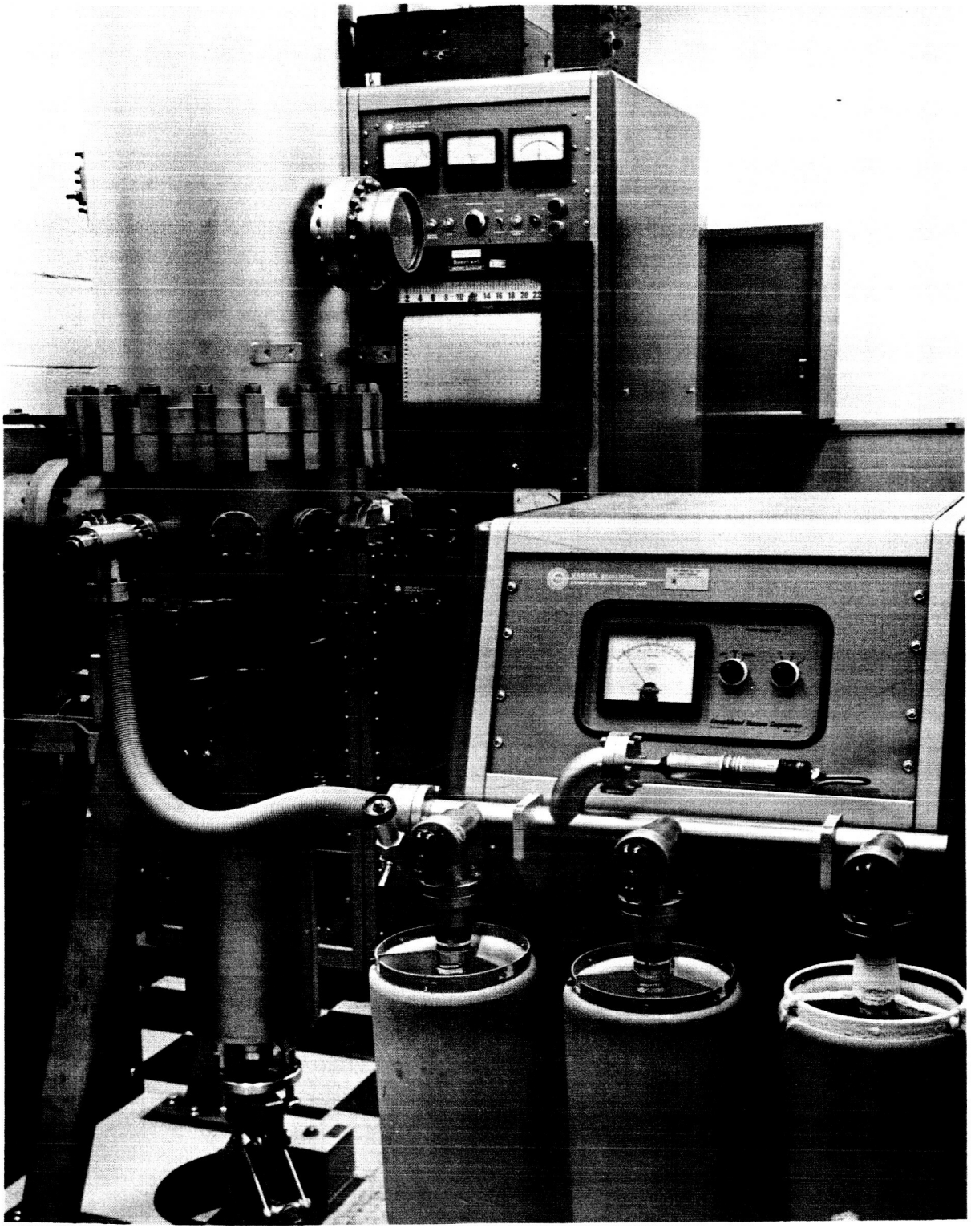


FIGURE 3 - Cryogenic Sorption Roughing Pump Attached to Vacuum System

atmospheric to less than 5 microns. The sputter ion pump is put into operation after the system is roughed to 5 microns or less. A bakeable valve isolates the chamber from the roughing system. After baking at 250°C, the base pressure of the system is less than 5 x 10^{-10} Torr. A typical pump down cycle for the system is shown in Table 1. Pressure is measured using a hot filament nude ionization gage located in the feed through sump.

Heat is applied to the test specimen by radiation from a tantalum resistance element 1-3/4 inch diameter x 6 inches long. Tantalum radiation shields and a water cooled copper cold wall assembly surround the heating element. Power to the heating element is controlled by a saturable reactor. Temperature is monitored and controlled by Pt-Pt13Rh thermocouples resistance welded to 1.5 mil Ta foil which is affixed to the gage length of the test specimen. The creep test specimen configuration is shown in Appendix I. A temperature gradient of less than 1°F was measured along the one inch gage length at 2400°F using thermocouples.

Creep deformation is determined by measuring the separation of fiducial lines applied to the ends of the specimen gage length. The strain measuring instrument for determining the linear dimensional change is shown in Figure 4. The instrument was procured from the Gaertner Scientific Company. Dimensional changes of 0.00005 inches can be measured with the instrument.

B. STARTING MATERIAL

One hundred pounds of unalloyed tantalum was purchased for alloy base for the melting of the balance of the two inch diameter consumable electrode melted ingots. The tantalum was purchased in the form of 1/4 inch thick plate and will be reduced to the required starting size by rolling. This completes procurement of all the starting material required for this contract effort.

C. 800 GRAM INGOTS

1. Melting

Twenty-three additional compositions were melted bringing the total number of 800 gram ingots melted to 45. The list of compositions melted during this report period is given in Table 2. Compositions NAS 34-49 were selected to investigate the effects of the addition and/or substitution of molybdenum and/or rhenium, zirconium, and nitrogen for tungsten, hafnium, and carbon respectively. A statistical design of one-quarter replication was utilized in selection of these compositions. A total of 9 a/o W + Hf was chosen for the base line for this series of compositions because ductile TIG welds were produced in sheet at the 9 a/o W + Hf level with carbon additions as high as 0.035 w/o.

TABLE 1 - Typical Pump-Down Time-Pressure History for Ultra-High Vacuum Creep Unit

Time (hrs.)	Pressure (Torr)	Remarks
0.0	760	Vacsorb #1 open
0.1	190	Vacsorb #2 opened - #1 closed
0.3	17×10^{-3}	Vacsorb #3 opened - #2 closed
0.35	9×10^{-3}	Ion pump on
0.45	5×10^{-3}	Bakeable valve closed
0.85	4×10^{-4}	(Ion pump started)
1.1	1.2×10^{-6}	System Baked at 250°C (480°F) for 14 Hours Maximum Pressure During Bakeout 5×10^{-5} Torr
15.0	---	Bakeout terminated
20.0	3×10^{-10}	System at room temperature

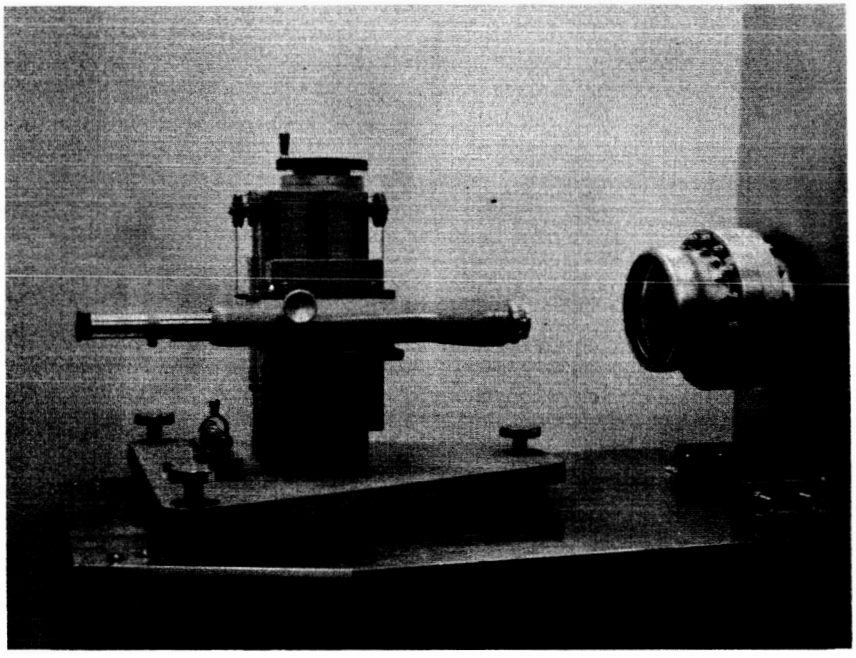


FIGURE 4 - Optical Strain Measuring Instrument

TABLE 2 - Compositions Melted as 800 Gram Ingots

Heat Number	W		Mo		Re		Hf		Zr		C		N	
	a/o	w/o	a/o	w/o	a/o	w/o	a/o	w/o	a/o	w/o	a/o	w/o	a/o	w/o
NAS-27	4.5	4.6	--	--	--	--	1.5	1.5	--	--	0.75	0.05	--	--
NAS-28	4.5	4.6	--	--	--	--	1.5	1.5	--	--	1.50	0.10	--	--
NAS-29	4.5	4.6	--	--	--	--	1.5	1.5	--	--	2.25	0.15	--	--
NAS-30	4.0	4.1	--	--	--	--	2.0	2.0	--	--	1.00	0.067	--	--
NAS-31	4.0	4.1	--	--	--	--	2.0	2.0	--	--	2.00	0.134	--	--
NAS-32	3.0	3.1	--	--	--	--	3.0	3.0	--	--	1.50	0.102	--	--
NAS-33	3.0	3.1	--	--	--	--	3.0	3.0	--	--	3.00	0.204	--	--
NAS-34	6.8	7.0	1.7	0.85	--	--	0.25	0.25	0.25	0.13	--	--	0.50	0.040
NAS-35	6.8	7.0	1.7	0.85	--	--	--	--	0.50	0.26	--	--	0.25	0.020
NAS-36	5.6	5.7	1.4	0.7	1.5	1.56	0.25	0.25	0.25	0.13	0.25	0.017	0.25	0.020
NAS-37	5.6	5.7	1.4	0.7	1.5	1.56	--	--	0.50	0.26	0.125	0.008	0.125	0.010
NAS-38	7.0	7.1	--	--	1.5	1.56	--	--	0.50	0.26	--	--	0.25	0.020
NAS-39	7.0	7.1	--	--	1.5	1.56	0.25	0.25	0.25	0.13	--	--	0.50	0.040
NAS-40	8.5	8.7	--	--	--	--	--	--	0.50	0.26	0.125	0.008	0.125	0.010
NAS-41	8.5	8.7	--	--	--	--	0.25	0.25	0.25	0.13	0.25	0.017	0.25	0.020
NAS-42	5.2	5.3	1.3	0.65	1.5	1.56	--	--	1.00	0.52	--	--	1.00	0.080
NAS-43	5.2	5.3	1.3	0.65	1.5	1.56	0.5	0.5	0.50	0.26	--	--	0.50	0.040
NAS-44	6.4	6.5	1.6	0.8	--	--	0.5	0.5	0.50	0.26	0.25	0.017	0.25	0.020
NAS-45	6.4	6.5	1.6	0.8	--	--	--	--	1.00	0.52	0.50	0.034	0.50	0.040
NAS-46	6.5	6.6	--	--	1.5	1.56	--	--	1.00	0.52	0.50	0.034	0.50	0.040
NAS-47	6.5	6.6	--	--	1.5	1.56	0.5	0.5	0.50	0.26	0.25	0.017	0.25	0.020
NAS-48	8.0	8.1	--	--	--	--	0.5	0.5	0.50	0.26	--	--	0.50	0.040
NAS-49	8.0	8.1	--	--	--	--	--	--	1.00	0.52	--	--	1.00	0.080

The technique used for non-consumable electrode melting of the 800 gram ingots was described in the second quarterly report[3] and the procedure was used without any further modifications. The rhenium, molybdenum, and zirconium additions were added as 0.010-0.020 inch thick sheet. Nitrogen additions to heats NAS 34-49 were made with a tantalum-nitrogen master alloy. The master alloy was prepared by melting 100 gram buttons of unalloyed tantalum under a partial pressure of nitrogen. The as-melted buttons were crushed by ball milling and classified by screening to obtain a particle size within the range of -60 to +200 mesh. A total of 1200 grams of nitrogen master alloy was melted and the yield of useable powder was approximately 600 grams. Analysis of six samples taken at random from the powder product gave an average nitrogen content of 0.89 w/o. The range for the six analyses was 0.86 to 0.91 w/o. The nitrogen master alloy powder was added to the melting charge in tantalum foil envelopes in a manner similar to that used for the TaC addition. Recovery of the interstitial additions was excellent. Results of carbon and nitrogen analysis on as-melted button compositions are given in Table 3. The excellent recovery of the interstitial addition verified the high reliability of the melting technique being used.

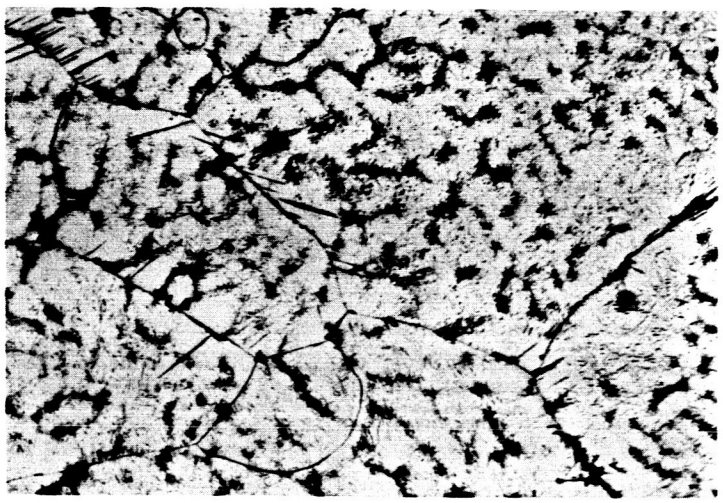
The non-homogeneous as-cast microstructure observed on the as-cast buttons which was discussed in the second quarterly report[3] was eliminated by a high temperature homogenization anneal. One hour treatments at 2000°C (3630°F), 2200°C (3990°F), and 2400°C (4350°F) were performed on as-cast samples taken from heat NAS-4 (Ta-14.6W-1.8Hf-0.12C). The as-cast microstructure was eliminated after one hour at 2400°C (4350°F) and was more than 95% eliminated after one hour at 2200°C (3990°F). Based upon this evaluation a one hour anneal at 2300°C (4170°F) was selected as the homogenizing treatment for all the alloys. The effects of the homogenizing treatment on NAS-4 are shown in Figure 5. It was deemed necessary to eliminate the microstructural inhomogeneity existing in the as-cast button because this variation was evident in the final sheet. The amount of work done on the button ingot during processing to sheet was insufficient to completely eliminate the constitutional segregation initially present in the surface layers.

A summary of metallographic observations on the button ingots melted during this period is listed in Table 4. The carbon-hafnium ratio apparently affects the morphology of the dispersed phase as illustrated in the photomicrographs shown in Figure 6. The tendency for a fine dispersed second phase at a hypostoichiometric C/Hf ratio and a platelet type second phase at a stoichiometric C/Hf ratio was observed in alloys containing 4.6W + 1.5Hf solute additions. A similar behavior was also observed on compositions containing 9 a/o W + Hf.

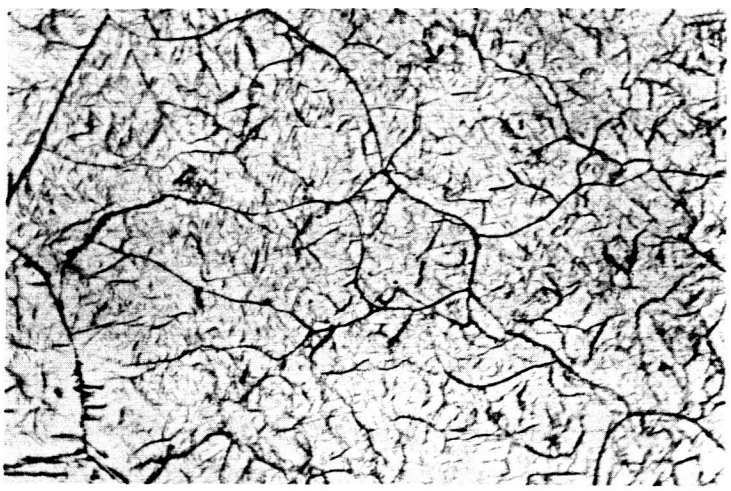
As-cast microstructures of compositions NAS-39 and NAS-42 are shown in Figure 7. Both of these compositions contain nitrogen. The microstructure of NAS-39 is essentially single phase and that of NAS-42 shows evidence of some second phase in the as-cast condition. The solubility of nitrogen in

TABLE 3 - Carbon and Nitrogen Analysis of Non-Consumable Electrode Melted 800 Gram Ingots

Heat No.	Carbon and/or Nitrogen Addition (w/o)		Analyzed Carbon and Nitrogen (w/o)			
			As-Cast		After 1 Hr. Anneal at 2300°C (4170°F)	
	C	N	C	N	C	N
NAS-14	0.09	---	0.073	---	---	---
NAS-34	---	0.04	---	0.034	---	0.018
NAS-35	---	0.02	---	0.022	---	0.013
NAS-36	0.017	0.02	0.016	0.017	0.013	0.012
NAS-37	0.008	0.01	---	---	0.0066	0.012
NAS-38	---	0.02	---	---	---	0.012
NAS-39	---	0.04	---	0.032	---	0.015
NAS-40	0.008	0.01	0.01	0.011	---	---
NAS-42	---	0.08	---	0.079	---	0.026



(a)



(b)

FIGURE 5 - Photomicrograph of NAS-4 (Ta-14.6W-1.8Hf-0.12C) (a) In the As-Cast Condition, and (b) After Annealing for One Hour at 2300°C (Electrolytic Etch) 150X

TABLE 4 - Summary of Metallographic Observations on Non-Consumable Melted 800 Gram Ingots

Heat No.	Composition (w/o)	DPH 30 Kg. Load		Microstructure
		As-Cast	As-Cast+1 Hr. at 2300°C (4170°F)	
NAS-27	4.6W-1.5Hf-0.05C	---	202	Rod type ppt. on sub-boundaries and random distribution
NAS-28	4.6W-1.5Hf-0.10C	---	235	Coarse Widmanstätten type ppt.-grain boundary carbides
NAS-29	4.6W-1.5Hf-0.15C	---	243	Fine Widmanstätten type ppt., random massive carbides, heavy grain boundary ppt.
NAS-30	4.1W-2.0Hf-0.067C	---	230	Widmanstätten type ppt., some grain boundary ppt.
NAS-31	4.1W-2.0Hf-0.134C	---	230	Fine Widmanstätten type ppt., massive grain boundary carbides
NAS-32	3.1W-3.0Hf-0.102C	---	230	Widmanstätten plus sub-boundary and grain boundary ppt.
NAS-33	3.1W-3.0Hf-0.204C	---	257	Fine Widmanstätten, massive grain boundary carbides
NAS-34	7.0W-0.25Hf-0.85Mo -0.13Zr-0.04N	341	318	Single Phase
NAS-35	7.0W-0.85Mo-0.13Zr -0.02N	300	288	Fine accicular type ppt., clean grain boundaries

TABLE 4 - Summary of Metallographic Observations on Non-Consumable Melted 800 Gram Ingots
(continued)

Heat No.	Composition (w/o)	DPH 30 Kg. Load		Microstructure
		As-Cast	As-Cast+1 Hr. at 2300°C (4170°F)	
NAS-36	5.7W-0.25Hf-0.017C -1.56Re-0.7Mo -0.13Zr-0.02N	368	322	Very fine spherical ppt.-widely dispersed
NAS-37	5.7W-0.008C-1.56Re -0.7Mo	319	303	Widely dispersed spherical ppt., appearance of sub-boundaries
NAS-38	7.1W-1.56Re-0.26Zr -0.02N	309	308	Single Phase
NAS-39	7.1W-0.25Hf-1.56Re -0.13Zr-0.04N	368	356	Single Phase
NAS-40	8.7W-0.008C-0.26Zr -0.01N	290	283	Fine spherical ppt., clean grain boundaries
NAS-41	8.7W-0.25Hf-0.017C -0.13Zr-0.02N	344	308	Fine spherical ppt.
NAS-42	5.3W-1.56Re-0.65Mo -0.52Zr-0.08N	470	482	Spherical ppt., agglomerated in clusters, randomly dispersed
NAS-43	5.3W-0.5Hf-1.56Re -0.65Mo-0.26Zr -0.04N	372	361	Fine spherical and accicular ppt., clean grain boundary
NAS-44	6.5W-0.5Hf-0.017C -0.8Mo-0.26Zr -0.02N	361	---	Almost single phase, fine ppt. in certain grains

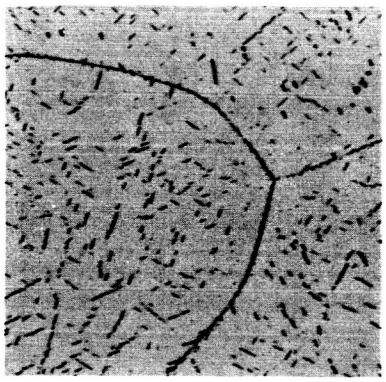
TABLE 4 - Summary of Metallographic Observations on Non-Consumable Melted 800 Gram Ingots
(continued)

Heat No.	Composition (w/o)	DPH 30 Kg. Load		Microstructure
		As-Cast	As-Cast+1 Hr. at 2300°C (4170°F)	
NAS-45	6.5W-0.034C-0.8Mo -0.52Zr-0.04N	388	---	Large amount of spherical ppt.
NAS-46	6.6W-0.034C-1.56Re -0.52Zr-0.04N	405	---	Same as NAS-45
NAS-47	6.6W-0.5Hf-0.017C -1.56Re-0.26Zr -0.02N	380	---	Generally single phase, some light dendritic ppt.
NAS-48	8.1W-0.5Hf-0.26Zr -0.04N	352	---	Single phase
NAS-49	8.1W-0.52Zr-0.08N	436	---	Duplex, areas of heavy ppt. and areas almost single phase

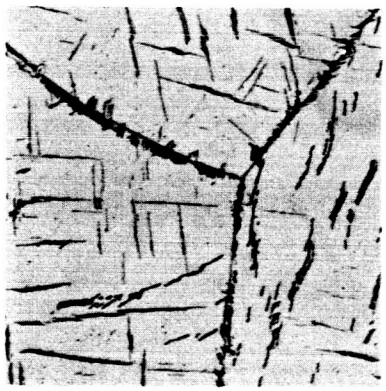
Remarks:

Heats NAS 27-43 as-homogenized

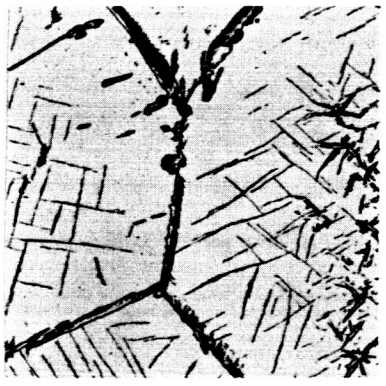
Heats NAS 44-49 as-melted



(a) C/Hf = 0.5

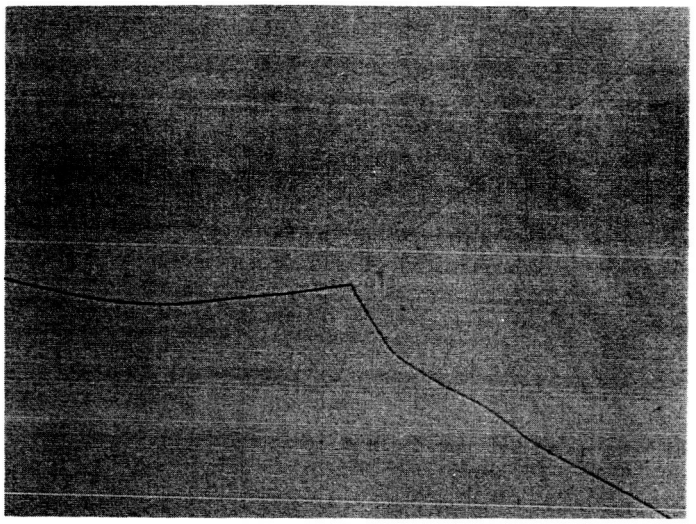


(b) C/Hf = 1.0

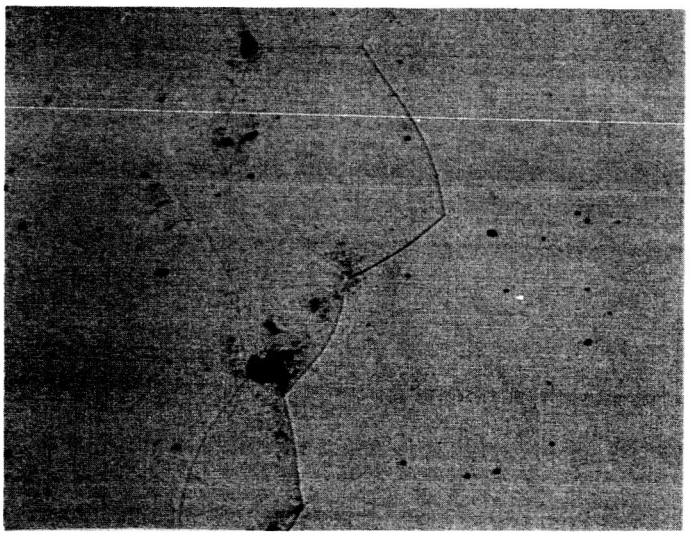


(c) C/Hf = 1.5

FIGURE 6 - Photomicrographs of a Ta-4.6W-1.5Hf Composition With (a) 0.05 w/o Carbon, (b) 0.10 w/o Carbon, and (c) 0.15 w/o Carbon, After Annealing As-Cast Buttons for One Hour at 2300°C (Electrolytic Etch) 150X



(a) NAS-39 (7.1W-1.56Re-0.25Hf-0.13Zr-0.04N)



(b) NAS-42 (5.3W-0.65Mo-1.56Re-0.52Zr-0.08N)

FIGURE 7 - As-Cast Microstructures of Re-Mo-Zr-N Modified Compositions (HNO_3-$NH_4F \cdot HF$ Etch) 100X

tantalum is approximately 50 times greater at 1800°C than that of carbon.[4]

The homogenizing treatment used for the button ingots was not amendable to the nitrogen containing buttons. Although the nitrogen was not lost during the melting operation, a significant amount of nitrogen was lost during the homogenization anneal. The data in Table 3 show that compositions containing up to 0.04 w/o nitrogen in the as-melted condition analyzed 0.015 w/o after the homogenization treatment. NAS-42 which had an addition of 0.08 w/o nitrogen analyzed 0.026 w/o after the homogenization treatment. The homogenization anneal was discontinued for compositions to which intentional nitrogen additions had been made. The data in Table 3 also indicate a slight loss of carbon occurred during the homogenization anneal.

The decarburization of molybdenum at temperatures of 2000°C (3630°F) in vacuum has been reported,[5,6] and decarburization of tantalum would also be expected to occur at elevated temperatures in vacuum. A 0.040 inch thick piece of sheet of NASV-4 (Ta-8W-2.75Hf-0.38Zr-0.05C) was heated for one hour at 2400°C (4350°F) and 10^{-5} Torr. One sample was wrapped in the tantalum foil and one sample was exposed bare. Carbon analysis showed that approximately 20% of the carbon was lost during the heat treatment. The results are shown below in Table 5.

TABLE 5 - Carbon Analysis on 0.040 Inch Sheet After Heating for One Hour at 2400°C (4350°F) and 10^{-5} Torr

Condition	Carbon (w/o)
As-Rolled	0.056
Wrapped with Ta Foil Annealed 1 Hour at 2400°C	0.039
Bare, Annealed 1 Hour at 2400°C	0.041

2. Primary Breakdown

The non-consumable melted ingots were coated with an oxidation resistant coating of Al-Si using the procedure described previously.[3] Heats NAS-27-33 and NAS-36, -37, and -38 were heated for forging in a globar furnace. The coated buttons were placed on a stainless steel pad and heated to temperature in an argon purged retort. The forging was done on the Dynapak Model 1220-C. It

appeared that the surface of the button ingot in contact with the stainless steel had reacted since during the forging operation numerous defects opened on this surface. Subsequently, all buttons were heated in an induction furnace, the specimens being supported on a molybdenum pedestal. An argon atmosphere was maintained during the heating cycle. This alleviated the surface problem somewhat, but did not eliminate it completely.

The results of the forging of button ingots NAS-27 through 49 are summarized in Table 6. The effect of the carbon addition on the forgeability of compositions containing 6 a/o W + Hf is shown in Figure 8. At the hyperstoichiometric C/Hf ratio, forgeability was generally fair to poor. This is not surprising since massive grain boundary carbide phase observed metallographically would be expected to seriously impair fabricability.

The as-forged buttons were conditioned by surface grinding, pickled, and any remaining defects were removed by hand grinding. The conditioned buttons were then annealed for one hour at 1650°C at $<10^{-5}$ Torr prior to rolling to sheet.

3. Secondary Working

The as-forged, conditioned, and annealed sheet bar was rolled to 0.060 inch thick sheet. The rolling results are in Table 7. Heats NAS 27 through 33 were rolled at ambient temperature. Compositions having a C/Hf ratio of stoichiometric and hypostoichiometric generally rolled satisfactorily with minor or no edge cracking. The compositions having a hyperstoichiometric C/Hf ratio experienced moderate to severe cracking during rolling and the yield of sheet was poor. Figure 9 is a graphic illustration of the 0.06 inch sheet obtained on heats NAS 27, 28, and 29 which again shows the effect of the carbon addition on fabricability.

The balance of conditioned sheet bars were given the initial breakdown rolling at 500°C (930°F) and finished from approximately 0.1 inch to 0.06 at ambient temperature. The initial warm breakdown rolling alleviated some of the cracking problems caused by poor shape of the conditioned sheet bar and also compositions which had marginal cold rolling properties. After rolling to 0.06 inch thick, samples were removed for determination of the one hour recrystallization temperature; the sheet was edge trimmed, cleaned, and annealed one hour at 1700°C (3090°F) at $<10^{-5}$ Torr. All compositions (NAS 27-43) were cold rolled from 0.06 to 0.04 inch thick sheet satisfactorily. All welding and mechanical property determinations were made on 0.04 inch thick sheet.

4. Recrystallization

The one hour recrystallization temperature was determined for

TABLE 6 - Summary of Forging Results

Heat No.	Forging Temp. (°C/°F)	As-Forged Thickness (in.)	Hardness (VPN)		Surface Condition
			As-Forged	As-Forged + 1 Hour At 1650°C (3000°F)	
NAS-27	1150/2100	0.259	---	186	Excellent
NAS-28	1150/2100	0.265	---	195	Good, localized surface failures, one side
NAS-29	1150/2100	0.270	---	202	Good, moderate edge cracking
NAS-30	1150/2100	0.263	---	192	Excellent
NAS-31	1150/2100	0.266	---	200	Minor edge cracking, localized surface failures, one side
NAS-32	1150/2100	0.266	---	193	Excellent
NAS-33	1150/2100	0.270	---	198	Moderate edge cracking
NAS-34	1500/2730	0.290	---	360	Excellent
NAS-35	1300/2370	0.275	---	300	Good
NAS-36	1150/2100	0.275	395	311	Good, surface defects, one side
NAS-37	1150/2100	0.270	382	300	Same as NAS-36
NAS-38	1150/2100	0.266	370	309	No edge cracking, minor surface defects
NAS-39	1600/2910	0.275	385	359	Excellent

TABLE 6 - Summary of Forging Results
(continued)

Heat No.	Forging Temp. (°C/°F)	As-Forged Thickness (in.)	Hardness (VPN)		Surface Condition
			As-Forged	As-Forged + 1 Hour At 1650°C (3000°F)	
NAS-40	1300/2370	0.275	---	281	Excellent
NAS-41	1300/2370	0.275	---	296	Excellent
NAS-42	1600/2910	0.275	474	383	Excellent
NAS-43	1500/2730	0.275	---	368	Good, edge cracking
NAS-44	1500/2730	0.280	376	---	Excellent, minor edge cracking
NAS-45	1500/2730	0.295	429	---	Good, moderate edge cracking, localized surface tears, one side
NAS-46	1500/2730	0.300	473	---	Moderate edge cracks, general surface tears, one side
NAS-47	1500/2730	0.280	411	---	Moderate edge cracks, general surface tears, one side
NAS-48	1500/2730	0.275	383	---	Good, minor edge cracks
NAS-49	1500/2730	0.285	---	----	Good, minor edge cracks

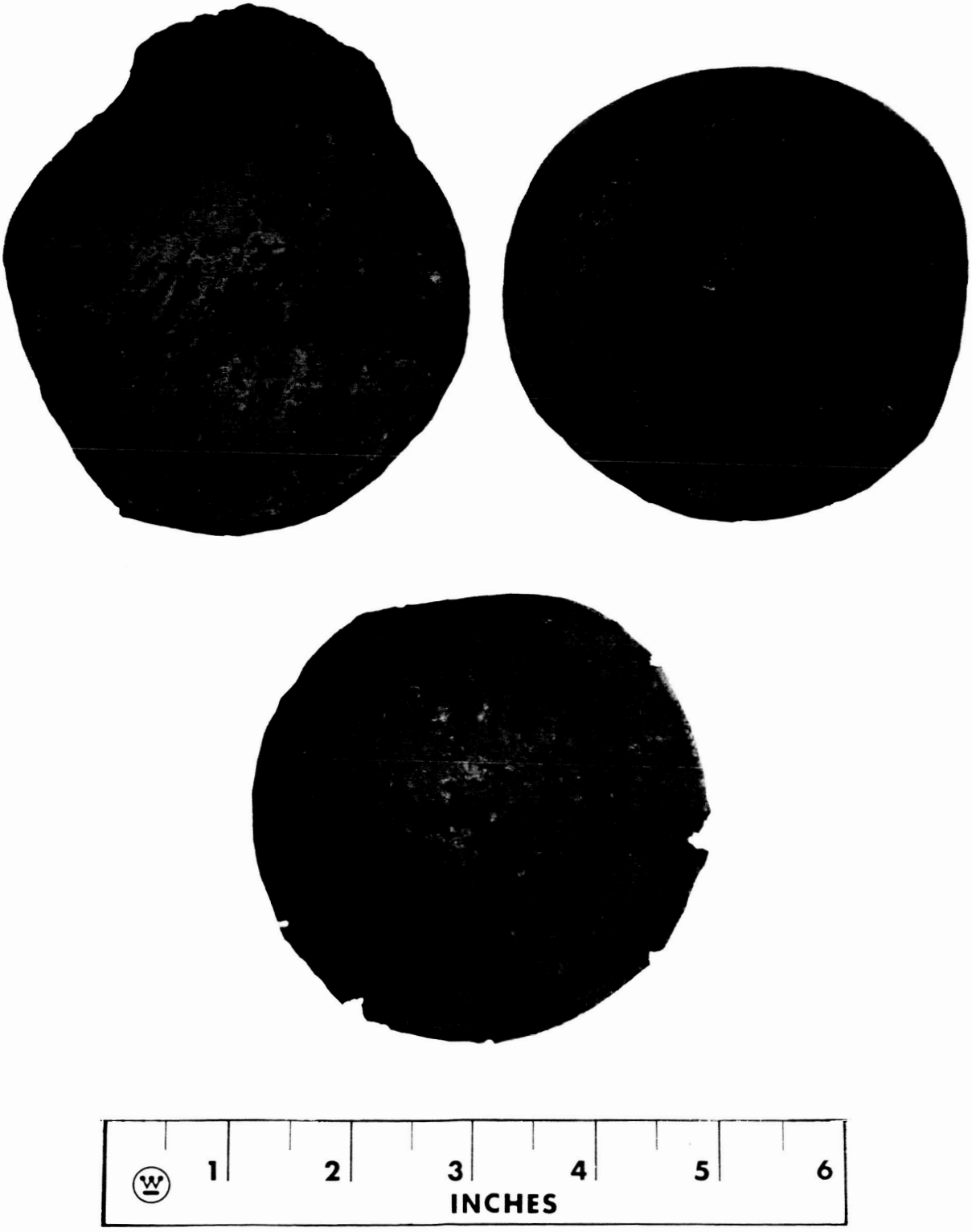


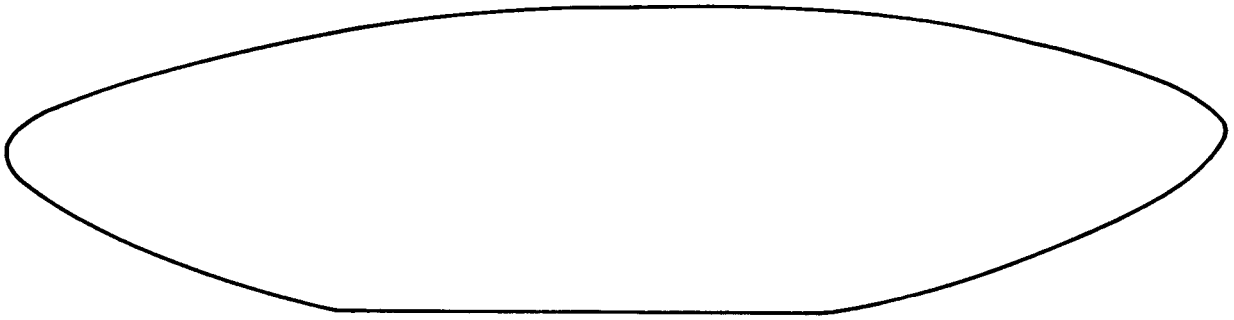
FIGURE 8 - Effect of Carbon on the Forgeability of a Ta-4.6W-1.5Hf Composition With (a) 0.05 w/o Carbon, (b) 0.10 w/o Carbon, and (c) 0.15 w/o Carbon

TABLE 7 - Rolling Data to 0.060 Inch Thick

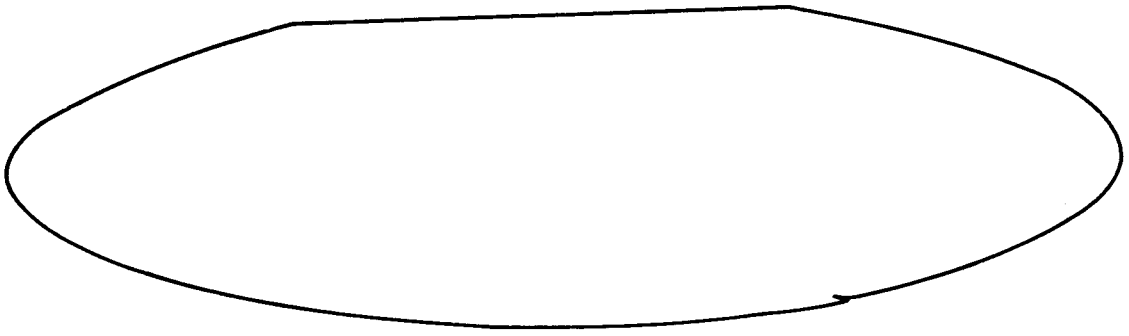
Heat No.	Starting Thickness (in.)	Total Reduction (%)	Initial Rolling Temp. (°C/°F)	Final Rolling Temp. (°C/°F)	As-Rolled Hardness DPH	Remarks
NAS-27	0.215	72.0	R.T.	R.T.	314	Excellent sheet
NAS-28	0.190	68.5	R.T.	R.T.	314	Excellent sheet
NAS-29	0.190	68.5	R.T.	R.T.	324	Moderate edge cracking, good sheet
NAS-30	0.237	74.5	R.T.	R.T.	311	Excellent sheet
NAS-31	0.190	68.5	R.T.	R.T.	318	Moderate edge cracking, good sheet
NAS-32	0.225	73.5	R.T.	R.T.	310	Minor edge cracking
NAS-33	0.215	72.0	R.T.	R.T.	329	Severe edge cracking, fair sheet
NAS-34	0.207	71.0	500/930	R.T.	410	Good quality sheet
NAS-35	0.215	72.0	500/930	R.T.	361	Same as NAS-34
NAS-36	0.183	68.0	R.T.	R.T.	381	Satisfactory, excellent strip

TABLE 7 - Rolling Data to 0.060 Inch Thick
(continued)

Heat No.	Starting Thickness (in.)	Total Reduction (%)	Initial Rolling Temp. (°C/°F)	Final Rolling Temp. (°C/°F)	As-Rolled Hardness DPH	Remarks
NAS-37	0.164	63.5	R.T.	R.T.	388	Same as NAS-36
NAS-38	0.199	70.0	R.T.	R.T.	400	Same as NAS-36
NAS-39	0.183	67.0	R.T.	320/610	433	Cracked at 0.167. Conditioned and rolled to 0.152, cracked reconditioned, rolled warm from 0.152.
NAS-40	0.213	72.0	500/930	R.T.	374	Same as NAS-34
NAS-41	0.231	74.0	500/930	R.T.	388	Same as NAS-34
NAS-42	0.210	71.5	R.T.	500/930	482	Cracked at 0.182, conditioned and rolled warm.
NAS-43	0.217	72.5	500/930	R.T.	420	Same as NAS-34



(a)



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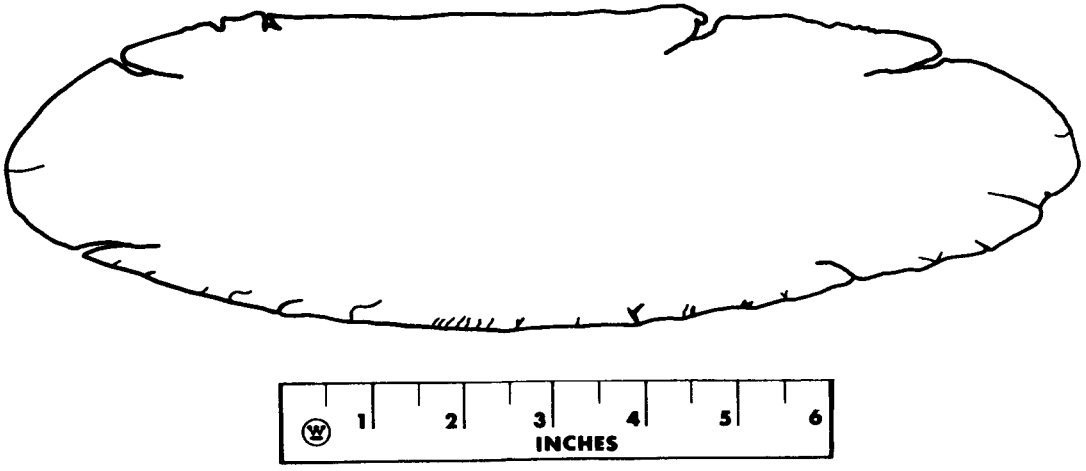


(c)

FIGURE 9 - Outline of As-Rolled 0.06 Inch Sheet of a Ta-4.6W-1.5Hf Alloy Composition With (a) 0.05 w/o Carbon, (b) 0.10 w/o Carbon, and (c) 0.15 w/o Carbon

compositions that were satisfactorily rolled to 0.06 inch thick sheet. Recrystallization was followed by means of metallographic examination and hardness measurements. The results are summarized in Table 8. Although the processing schedule was essentially similar for all compositions, minor variations, necessitated to produce maximum yield of useable material, permits only qualitative comparisons concerning the effects of the alloy additions. Increasing the W + Hf content from 6 a/o to 9 a/o raised the temperature at which equiaxed grains began to form by approximately 200°C (360°F). An additional increase in the solute content to 12 a/o W + Hf did not cause a corresponding increase in this temperature. Carbon additions appeared to retard the onset of recrystallization, but the effectiveness of the carbon addition appears related to the amount of hafnium present.

The hardness minima observed in compositions containing 9 and 12 a/o W + Hf occurred prior to the complete formation of equiaxed grains. The increase in room temperature hardness observed after heating above 1400°C (2550°F) is attributed to resolutioning of the dispersed carbide phase. Grain coarsening was observed after heating for one hour at 1800°C (3270°F) and above for compositions containing 9 and 12 a/o W + Hf.

The substitution and/or additions of the Mo, Re, Zr, and N to the 9 a/o W + Hf compositions had a significant effect on the recrystallization temperature. NAS-42 exhibited a wrought microstructure after being heated for one hour at 1600°C. This is a 300-400°C (540-720°F) increase in the recrystallization temperature as determined by metallographic examination. A very fine dispersed second phase was observed in the wrought NAS-42 which disappeared as equiaxed grains were forming. The cooling rate from 1700°C (3090°F) and above was apparently fast enough to suppress precipitation.

Figure 10 is a plot of room temperature hardness versus the one hour annealing temperature for selected compositions. The shapes of the various curves cannot all be explained at this time, but the subtle effects of the Mo, Re, Zr, and N additions on the hardness and recrystallization behavior of the 9 a/o W + Hf compositions are evident.

5. Weldability

The ductile-brittle bend transition temperature of as-welded specimens is the criterion for evaluating the effects of compositionsal variations on weldability. Ductile weld joints are a prime requisite for fabrication of reliable sheet and tubing components for space power applications. Tungsten electrode welding in an inert gas atmosphere (TIG) and electron beam (EB) welding techniques were used. TIG welded bend specimens were prepared by butt welding suitably prepared 1/2 inch wide x 5 inches long strips clamped in a stainless steel fixture with copper bar clamps. The

TABLE 8 - Summary of One Hour Recrystallization Results on 0.06 Inch Thick Sheet Reduced Approximately 70%

Heat No.	Composition	As-Rolled	Annealing Temperature, °C/°F							
			1200	1300	1400	1500	1600	1700	1800	2000
			2190	2370	2550	2730	2910	3090	3270	3630
NAS-6	8.2W-1.0Hf-0.035C	357 W	328 W	280 W	237 RB	255 RP	250 R	247 R	252 R	249 R
NAS-7	8.2W-1.0Hf-0.070C	371 W	343 W	301 W	239 RB	249 RP	257 RP	249 R	249 R	256 R
NAS-8	8.2W-1.0Hf-0.100C	390 W	344 W	305 W	243 RB	256 RP	243 RP	260 R	252 R	260 R
NAS-20	8.5W-0.7Hf-0.045C	363 W	323 W	292 RB	232 RB	250 R	248 R	239 R	248 R	247 R
NAS-24	8.5W-0.7Hf-0.070C	375 W	305 W	265 RB	230 RP	237 R	247 R	242 R	249 R	253 R
NAS-21	8.6W-0.53Hf-0.020C	337 W	306 W	259 RB	226 RP	240 R	239 R	246 R	243 R	249 R
NAS-22	8.6W-0.53Hf-0.035C	352 W	323 W	247 RB	232 RP	240 R	238 R	236 R	244 R	243 R

Remarks:
W - Wrought
RB - Formation of equiaxed grains 50%
RP - Formation of equiaxed grains 50%
R - Formation of equiaxed grains 98%
S - Single Phase

TABLE 8 - Summary of One Hour Recrystallization Results on 0.06 Inch Thick Sheet Reduced Approximately 70%
(continued)

Heat No.	Composition	As-Rolled	Annealing Temperature, °C/°F							
			1200	1300	1400	1500	1600	1700	1800	2000
			2190	2370	2550	2730	2910	3090	3270	3630
NAS-23	8.6W-0.53Hf-0.050C	347 W	318 W	255 RB	223 RB	228 R	237 R	237 R	240 R	241 R
NAS-16	11.3W-0.92Hf-0.06C	431 W	356 W	---	272 RP	---	300 R	---	295 R	290 R
NAS-27	4.6W-1.5Hf-0.05C	296 W	222 RB	---	186 R	---	196 R	198 R	213 R	209 R
NAS-28	4.6W-1.5Hf-0.10C	314 W	210 RB	---	197 R	---	196 R	208 R	211 R	211 R
NAS-29	4.6W-1.5Hf-0.15C	324 W	214 RB	---	205 R	---	199 R	205 R	208 R	219 R
NAS-30	4.1W-2.0Hf-0.067C	311 W	203 RB	---	192 R	---	194 R	200 R	204 R	209 R
NAS-31	4.1W-2.0Hf-0.134C	318 W	214 RB	---	203 R	---	205 R	207 R	208 R	218 R
NAS-32	3.1W-3.0Hf-0.102C	310 W	241 W	---	194 R	---	196 R	196 R	203 R	214 R
NAS-33	3.1W-3.0Hf-0.204C	329 W	250 W	---	209 R	---	206 R	207 R	210 R	217 R

TABLE 8 - Summary of One Hour Recrystallization Results on 0.06 Inch Thick Sheet Reduced Approximately 70%
(continued)

Heat No.	Composition	As-Rolled	Annealing Temperature, °C/°F							
			1200	1300	1400	1500	1600	1700	1800	2000
			2190	2370	2550	2730	2910	3090	3270	3630
NAS-34	7.0W-0.25Hf-0.85Mo-0.13Zr -0.04N	410 W	332 W	---	---	---	288 R,S	---	275 R,S	---
NAS-35	7.0W-0.85Mo-0.26Zr-0.02N	361 W	358 W	---	---	---	299 R	---	303 R	---
NAS-36	5.7W-0.25Hf-1.56Re-0.7Mo -0.13Zr-0.017C-0.02N	381 W,S	367 W,S	---	317 RB,S	---	318 R,S	292 R,S	316 R,S	323 R,S
NAS-37	5.7W-1.56Re-0.7Mo-0.26Zr -0.008C-0.01N	388 W,S	358 W,S	---	315 RB,S	---	306 R,S	303 R,S	309 R,S	298 R,S
NAS-38	7.1W-1.56Re-0.26Zr-0.02N	400 W,S	362 W,S	---	302 RB,S	---	278 RP,S	288 RP,S	283 RP,S	282 R,S
NAS-39	7.1W-0.25Hf-1.56Re-0.13Zr -0.04N	433 W,S	414 W,S	---	357 RB,S	---	346 RB,S	341 RP,S	338 RP,S	305 R,S
NAS-40	8.7W-0.008C-0.26Zr-0.01N	374 W	386 W	---	---	---	320 RB	---	336 R	---
NAS-41	8.7W-0.25Hf-0.017C-0.13Zr -0.02N	388 W	375 W	---	---	---	337 RP,S	---	310 R,S	---
NAS-42	5.3W-1.56Re-0.65Mo-0.52Zr -0.08N	482 W	414 W	---	368 W	---	346 W	392 RB	392 RP,S	355 R,S
NAS-43	5.3W-0.5Hf-1.56Re-0.65Mo -0.26Zr-0.04N	420 W	435 W	---	---	---	---	---	372 R,S	---

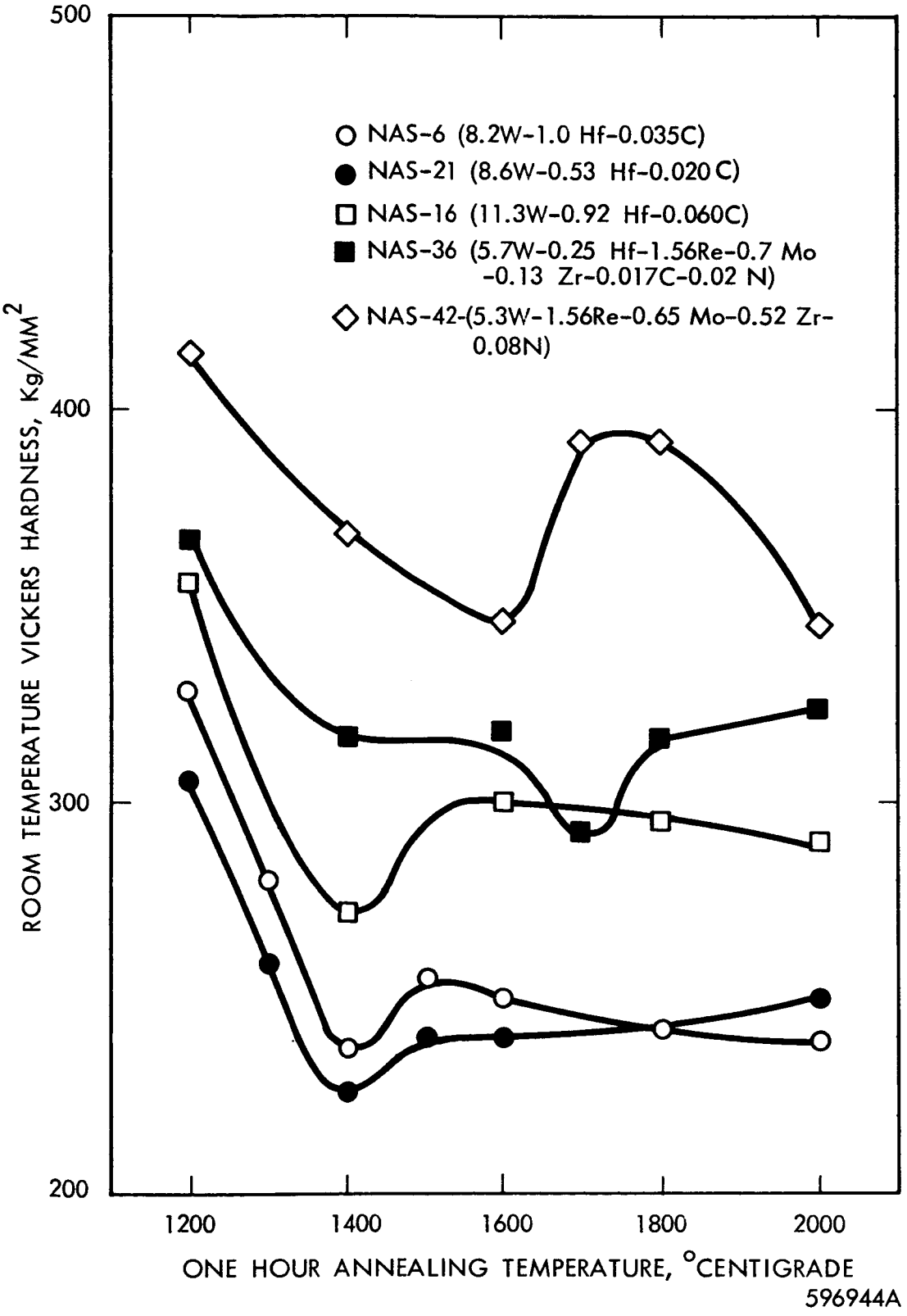


FIGURE 10 - Room Temperature Hardness of 0.06 Inch Sheet, (~70% Prior Cold Work) After One Hour Annealing Treatments

welding conditions were standardized and used for all compositions and are listed in Table 9.

TABLE 9 - TIG Welding Parameters

Welding Current	100 amps
Welding Speed	15 inches per minute
Arc Gap	0.06 inches
Jaw Spacing	3/8 inch
Electrode Diameter	3/32 inch

The welding chamber for TIG welding was evacuated to 10^{-5} Torr and backfilled with helium. The oxygen and moisture content in the weld chamber was continuously monitored during welding. Oxygen was nominally less than 5 ppm and water vapor was nominally 1 ppm during welding.

EB welding was done in the 2 K.W. Zeiss Electron Beam Welder shown in Figure 11. Bead-on-sheet welds with 100% penetration were the requirements for these welds. The weld chamber was evacuated to 5×10^{-6} Torr before welding. The weld bead was made on 1/2 inch wide x length x 0.040 inch thick strip. All welds were made on material in the as-rolled condition. Data for the EB welds are listed in Table 10.

Bend specimens were prepared with the weld bead transverse to the bend axis. The bend specimens were 0.040 inches thick x 12t wide x 24t long, with the top of the weld bend the tension side during testing. Other fixed test parameters were a punch radius of 0.071 inch ($\sim$2t), 1 inch per minute ram speed, and a span width of 15t (0.6 inches). The equipment used for bend testing is shown in Figure 12. A thermocouple attached to the ram and in contact with the specimen is used for controlling the selected test temperature. Temperatures below room temperature are obtained with a liquid nitrogen cryostat and above room temperature with a hot air blower. The ductile-brittle bend transition temperature is defined as the lowest temperature at which a full 90° bend (after springback) can be made without any failure. The ductile-brittle transition temperature was normally determined within 25-50°F, but lack of material for some compositions did not allow this close a definition of the transition temperature for all the tests which are summarized in Table 11.

The initial series of EB welds were made using a welding speed of 50 ipm. However, at this welding speed, the bend test results were not sufficiently discriminatory as evidenced by the fact that the transition

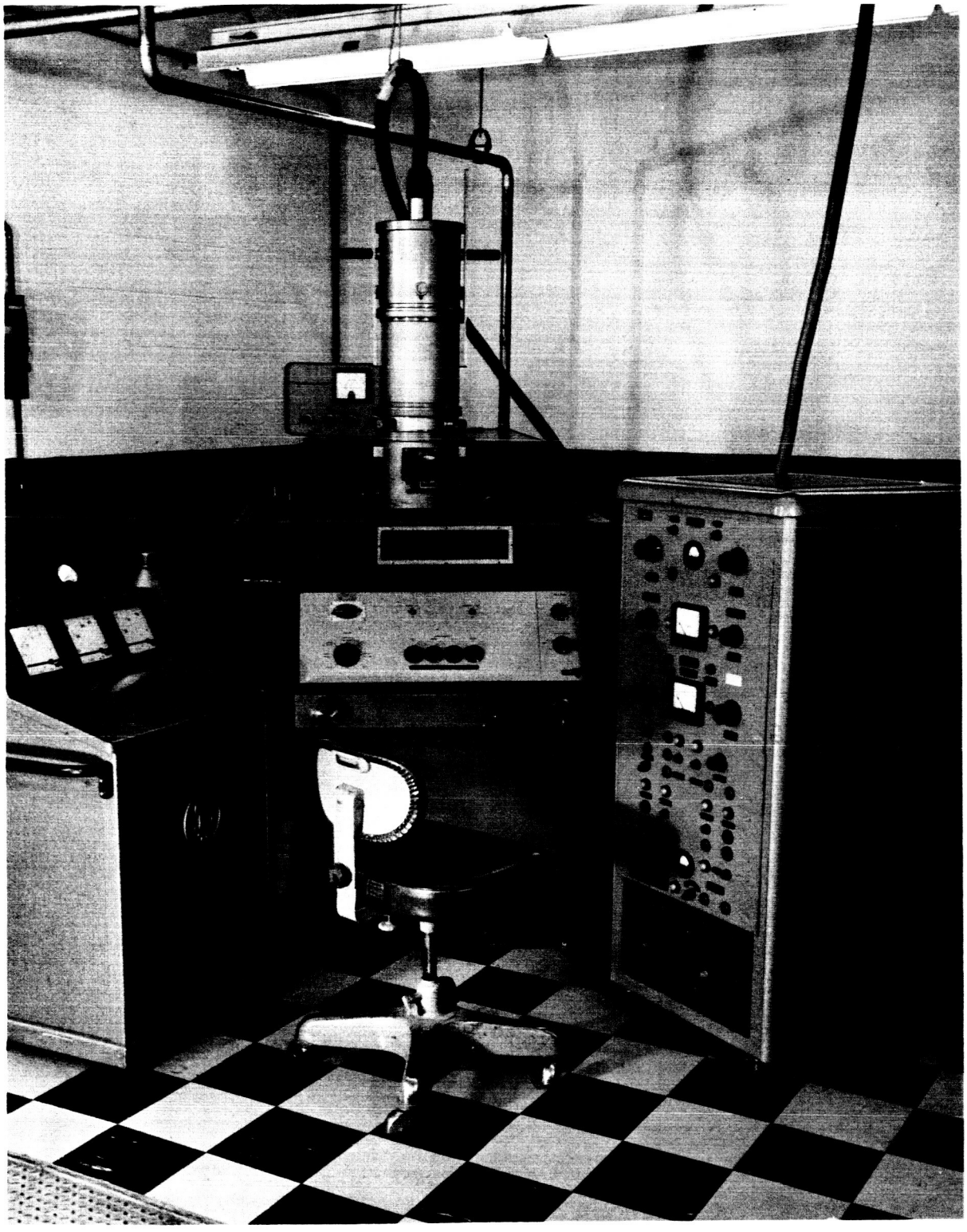


FIGURE 11 - 2KW-Zeiss Electron Beam Welder

TABLE 10 - Electron Beam Welding Data

Heat No.	Pressure x 10^{-6} Torr	Potential K.V.	Electron Current (milliamps)	Welding Speed[a] (inches per minute)
NAS-6	---	100	6.25	50
NAS-7	---	100	6.25	50
NAS-8	---	100	6.25	50
NAS-8	7.6	100	5.40	25
NAS-20	6.0	100	5.40	25
NAS-20[b]	12.0	100	6.25	50
NAS-24	7.5	100	6.25	50
NAS-21	8.5	100	6.25	50
NAS-22	9.0	100	6.25	50
NAS-23	---	100	6.25	50
NAS-13	5.0	100	5.40	25
NAS-14	4.0	100	5.40	25
NAS-16	5.0	100	5.40	25
NAS-18[b]	4.0	100	5.40	25
NAS-1	10.0	100	5.40	25
NAS-27	4.8	100	5.80	25
NAS-28	4.8	100	5.80	25
NAS-29	4.8	100	5.80	25
NAS-30	4.4	100	5.80	25
NAS-31	4.4	100	5.80	25
NAS-32	4.4	100	5.80	25
NAS-33	1.6	100	6.00	25
NAS-34	2.5	100	6.00	25
NAS-36[b]	4.0	100	5.40	25
NAS-37	5.0	100	5.40	25
NAS-38	5.0	100	5.40	25
NAS-41	2.5	100	6.00	25
NAS-43	2.5	100	6.00	25

(a) 0.025" Transverse Deflection of Beam

(b) Incomplete Penetration

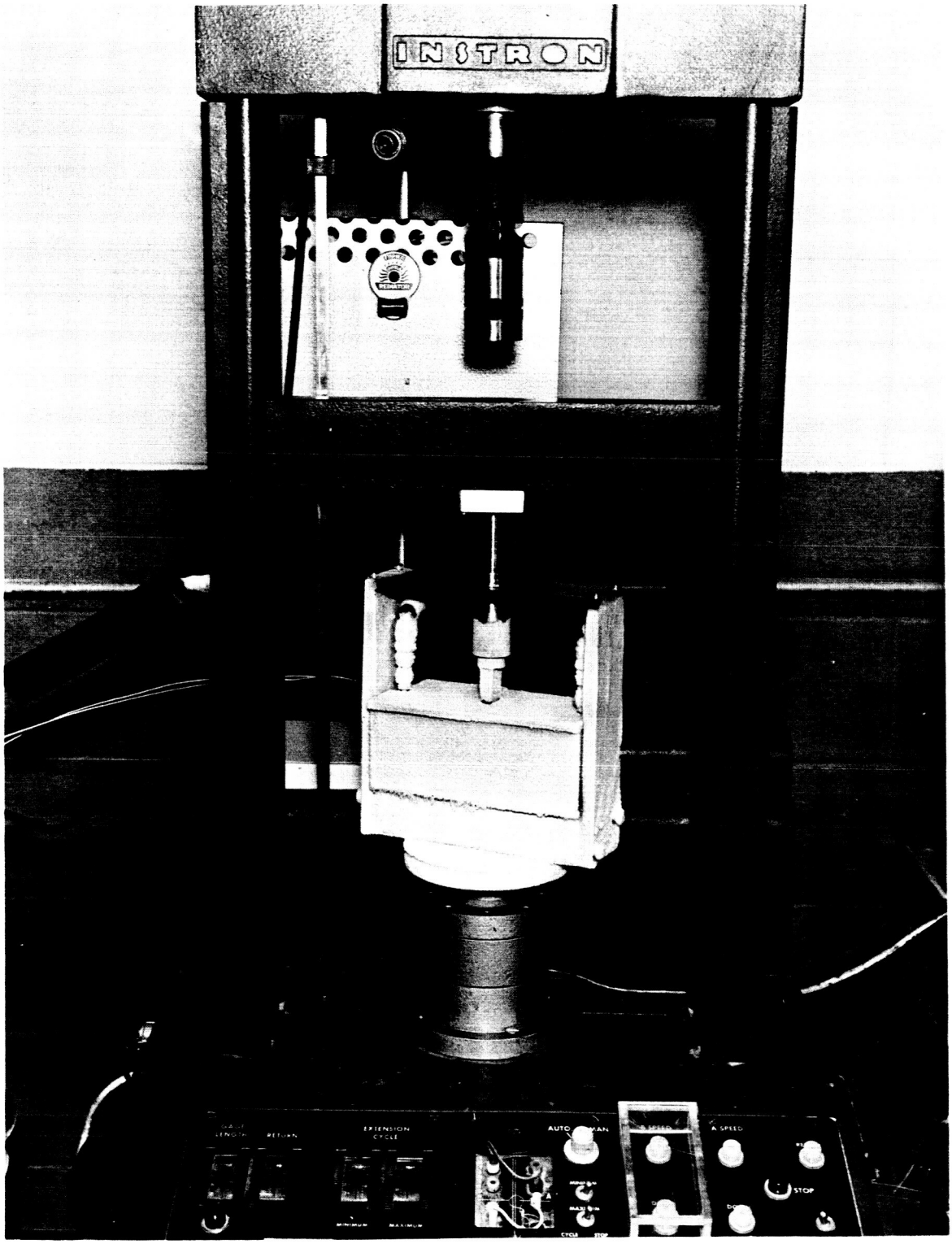


FIGURE 12 - Bend Test Equipment

TABLE 11 - Ductile-Brittle Transition Temperature for TIG and EB Welded Specimens Tested in Bending

Heat No.	Composition (w/o)	DBTT* (°F)		
		TIG Welded	EB Welded 25 ipm	EB Welded 50 ipm
NAS-6	8.2W-1.0Hf-0.035C	-50		-250
NAS-7	8.2W-1.0Hf-0.070C	> +300		
NAS-8	8.2W-1.0Hf-0.100C	> +300	+300	-100
NAS-20	8.5W-0.7Hf-0.045C	> +300	R.T.	-200
NAS-24	8.5W-0.7Hf-0.070C	> +300		-150
NAS-21	8.6W-0.53Hf-0.020C	-320		< -320
NAS-22	8.6W-0.53Hf-0.035C	> +300		-250
NAS-23	8.6W-0.53Hf-0.050C	> +300		-250
NAS-13	11.0W-1.33Hf-0.045C	---	+500	---
NAS-14	11.0W-1.33Hf-0.09C	---	+350	---
NAS-16	11.3W-0.92Hf-0.06C	> +500	+550	---
NAS-18	11.5W-0.70Hf-0.05C	---	0	---
NAS-1	14.6W-1.8Hf	---	+175	---
NAS-27	4.6W-1.5Hf-0.05C	< -320	< -320	---
NAS-28	4.6W-1.5Hf-0.10C	R.T.	-175	---
NAS-29	4.6W-1.5Hf-0.15C	> +500	R.T.	---
NAS-30	4.1W-2.0Hf-0.067C	> +500	-320	---
NAS-31	4.1W-2.0Hf-0.134C	> +300	R.T.	---
NAS-32	3.1W-3.0Hf-0.102C	> +500	-25	---
NAS-33	3.1W-3.0Hf-0.204C	> +400	> +500	---
NAS-34	7.0W-0.25Hf-0.85Mo-0.13Zr -0.04N	---	-50	---
NAS-36	5.7W-0.25Hf-1.56Re-0.7Mo -0.13Zr-0.017C-0.02N	-200	-275	---
NAS-37	5.7W-1.56Re-0.7Mo-0.26Zr -0.008C-0.01N	+105	R.T.	---
NAS-38	7.1W-1.56Re-0.26Zr-0.02N	-100	-225	---
NAS-41	8.7W-0.25Hf-0.13Zr-0.017C -0.02N	---	-100	---
NAS-43	5.3W-1.56Re-0.65Mo-0.5Hf -0.26Zr-0.04N	---	+200	---
NAS-39	7.1W-0.25Hf-1.56Re-0.13Zr -0.04N	+150	---	---

* Temperature for full 90° bend without failure

temperature was only increased approximately 170°F when the carbon content was increased from 0.02 w/o to 0.10 w/o for compositions containing 9 a/o W + Hf. EB welds were made at 25 ipm and the EB bend test results were more comparable to those obtained on the TIG welded specimens. The ductile-brittle transition temperature for TIG welded specimens was extremely sensitive to the carbon content. Figure 13 is a plot of the weld bend transition temperature as affected by the carbon content for a base composition of 9 a/o W + Hf. The pronounced effect of welding speed on bend ductility is strongly indicative of a carbide precipitation effect.

Metallographic examination showed that the heat affected zone was single phase for NAS-6 and NAS-22 (0.035C) and NAS-20 (0.045C) and contained a precipitate on NAS-7 (0.07C) and NAS-8 (0.10C). This suggests that the carbon solubility in tantalum as reported by Pochon et.al.[7] of 0.02 a/o at 2800°C may be too low. The value reported by Pochon et.al. is in disagreement with that reported by Storms[8] of approximately 0.5 a/o carbon at 2902°C (5256°F).

Hardness traverses of a series of the TIG welded specimens are shown in the plot in Figure 14. The shape of the plots indicates that the high hardness in the heat affected zone is attributable to the carbon retained in solution during the rapid quench occurring during cooling after welding.

Another interesting result is that the weld bend transition of TIG welds is apparently affected by the C/Hf ratio. NAS-6 (C/Hf = 0.5) is weld ductile at -50°F while NAS-22 (C/Hf = 1.0) has a ductile-brittle transition temperature above +300°F. Both compositions have a carbon content of 0.035 w/o. This same behavior, however, was not observed in the EB welded specimens.

6. Mechanical Properties

a. Hot Hardness: Elevated temperature hardness measurements are summarized in Table 12. Hardness measurements were made on 0.040 inch thick material in the as-rolled condition. Hot hardness measurements are extremely useful in establishing trends and for screening quickly a large number of alloy compositions. The hardness value is also indicative of the tensile strength although no insight as to fracture characteristics can be deduced solely from the hardness data. The fraction of room temperature hardness retained at the elevated temperature is indicative of the deterioration of the mechanism(s) responsible for the strength levels obtained at the lower temperature. Hardness data at 1100°C (2010°F) and higher indicate that strain hardening is not a very effective strengthening mechanism for the alloy compositions tested. NAS-36 with a hardness of 217 DPH at 1100°C (2010°F) is comparable to the hardness exhibited by T-111 at room temperature.

b. Tensile Tests: Tensile data were obtained at room and at

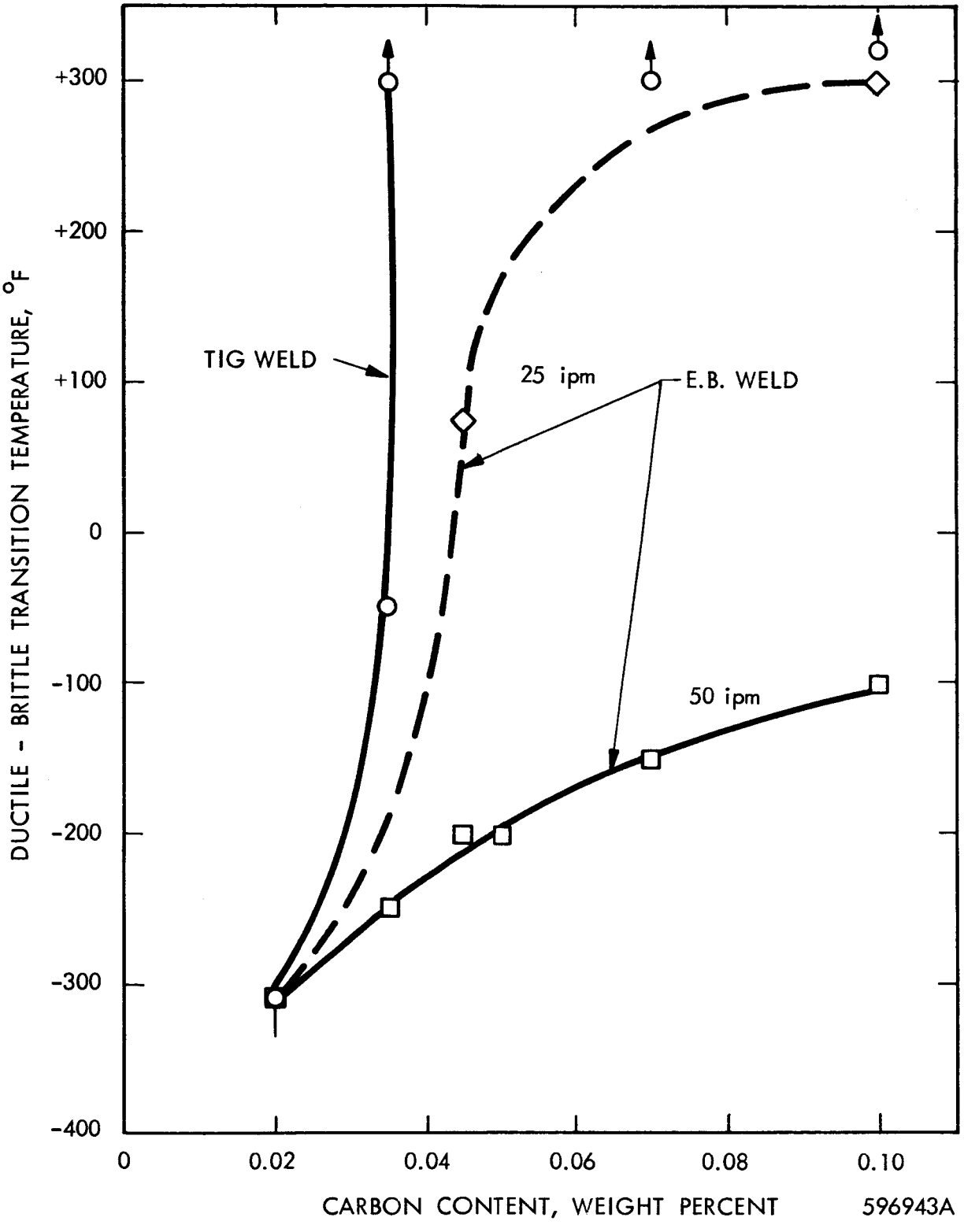


FIGURE 13 - Effect of Carbon on Ductile-Brittle Transition Temperature of Compositions Containing 9 a/o W + Hf

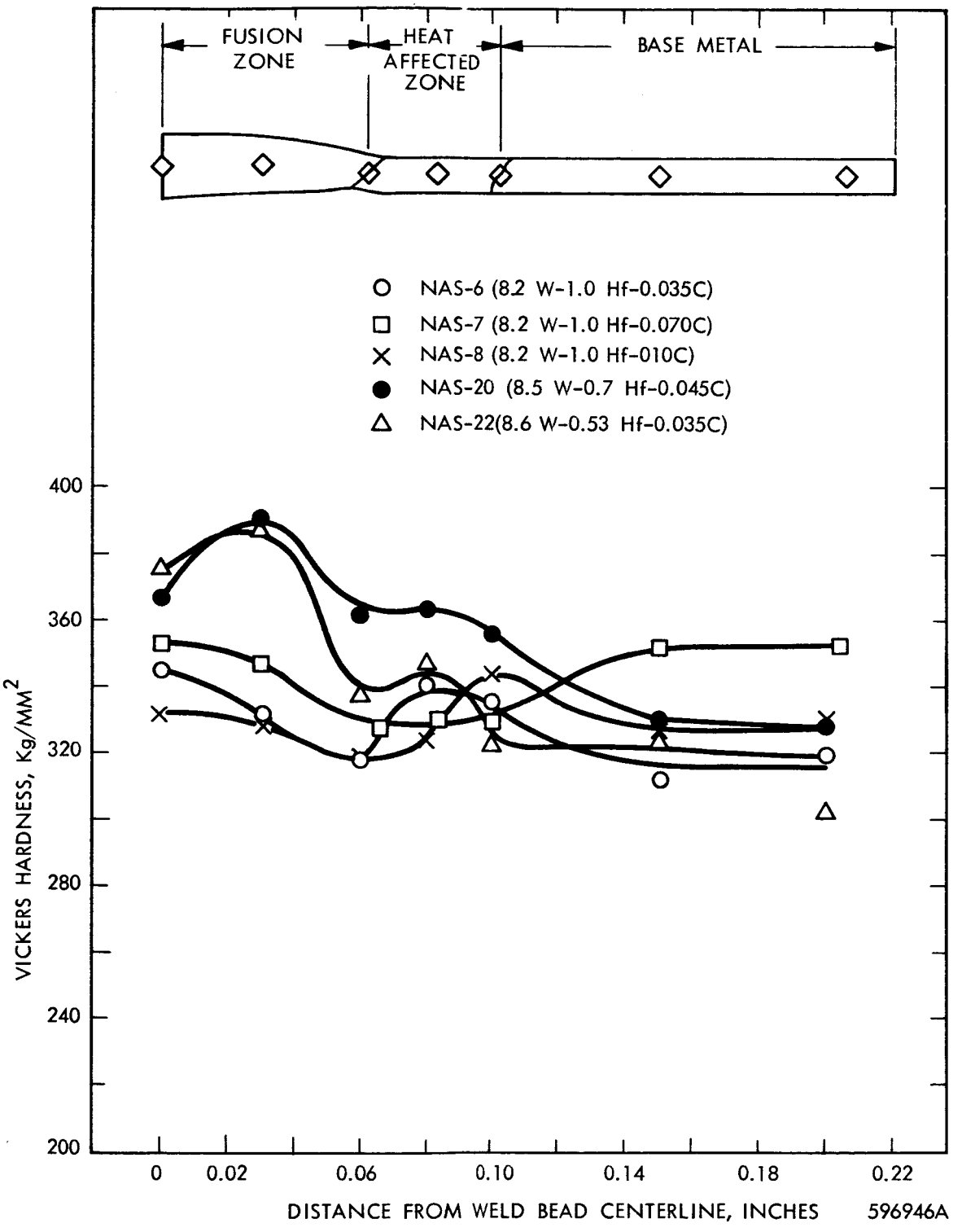


FIGURE 14 - Hardness Traverse of As-Welded Specimens

TABLE 12 - DPH Hardness[a] As a Function of Temperature 800 Gram Ingot Compositions As-Rolled 0.040 Inch Sheet

Heat No.	R.T.	Temperature, °C/°F			
		815/1500	1100/2000	1200/2200	1315/2400
T-111[c]	230	--	138 (.60)	120 (.52)	99 (.43)
NAS-1	341	286 (.84)[b]	--	--	--
NAS-2	309	289 (.94)	--	--	--
NAS-3	426	306 (.72)	--	--	--
NAS-5	392	253 (.64)	--	--	--
NAS-6	298	239 (.80)	175 (.59)	--	115 (.39)
NAS-7	331	267 (.81)	205 (.62)	--	120 (.36)
NAS-8	346	274 (.79)	194 (.56)	--	120 (.35)
NAS-9	385	293 (.76)	--	--	--
NAS-10	435	281 (.64)	--	--	--
NAS-12	---	295	--	--	--
NAS-13	365	255 (.70)	--	--	--
NAS-14	342	296 (.86)	--	--	--
NAS-18	358	273 (.76)	--	--	--
NAS-20	314	261 (.83)	210 (.67)	--	127 (.40)
NAS-21	295	241 (.82)	176 (.60)	--	99 (.33)
NAS-22	301	254 (.84)	175 (.58)	--	99 (.33)
NAS-23	315	252 (.80)	190 (.60)	--	100 (.32)
NAS-24	315	261 (.83)	190 (.60)	--	100 (.32)
NAS-25	355	273 (.77)	--	--	--
NAS-26	---	290	--	--	--
NAS-36	349	--	217 (.62)	137 (.39)	--
NAS-37	376	287 (.76)	192 (.51)	139 (.37)	--
NAS-38	353	253 (.72)	142 (.40)	110 (.31)	--
NAS-39	372	280 (.75)	198 (.53)	139 (.37)	--
NAS-42	443	299 (.68)	202 (.46)	136 (.31)	--

(a) 30 Kg. load for R.T. hardness measurements. 2-1/2 KG. for elevated temperature measurements.
(b) Fraction of room temperature hardness in parentheses.
(c) T-111 data reported by Ammon and Begley[11], material tested in annealed condition, one hour at 1650°C (3000°F)

temperature and are summarized in Table 13. The more detailed elevated tensile data were obtained on compositions which exhibited good weld bend ductility. The tensile properties of NAS-6 and NAS-21 at 2400°F are both comparable to the tensile strength of T-222 at 2400°F which is reported[9] as 58,800 psi ultimate with a 38,100 psi yield strength. The tensile strength of NAS-6 and NAS-21 at 2400°F is due primarily to dispersed phase strengthening. NAS-6 and NAS-21 contain 9 a/o W + Hf while T-222 contains a total of 12 a/o W + Hf.

Significant room temperature strength increases were obtained at the 9 a/o substitutional solute level for the Mo, Re, Zr, and modified compositions (NAS 36, 38, 39, and 42). Interactions of the Mo, Zr, Re, and additions and/or substitutions to the Ta-W-Hf-C system are being investigated in some detail and are producing some interesting effects.

7. Creep Results

Creep results obtained at 10^{-8} Torr are in Table 14. The initial test on NAS-7 was conducted on material in the as-worked condition. The final cold reduction of 33% on the solution annealed material was primarily for the purpose of increasing the dislocation density to furnish nucleating sites for precipitation during creep testing. However, the test temperature was in the recrystallization range, thereby causing accelerated creep. This behavior has been observed by other investigators and is described by Perryman.[10] Subsequently all specimens were annealed one hour at 1650°C (3000°F) prior to testing. The decrease in creep rate for NAS-7 was significant. NAS-6 exhibited good creep properties at 1316°C (2400°F) and 15,000 psi exhibiting a total strain of 1.35% in 172 hours. One of the best commercially available columbium base alloys (FS-85) tested under comparable pressure conditions exhibited 3.35% elongation after 300 hours at 1316°C (2400°F) and a stress level of 4,000 psi. NAS-6 at 1316°C (2400°F) is approximately 3.5 times stronger in creep. The creep data obtained by Hall[2] are the only published long time creep data with which reasonable comparisons can be made To date most creep data in columbium and tantalum base alloys have been obtained in oil diffusion pumped systems, usually at pressures of 10^{-6} Torr or higher and it is probable that contamination of the test specimen may have occurred.

NAS-8 in the as-worked condition, tested in an oil diffusion pumped system equipped with a liquid nitrogen cold trap to retard backstreaming of diffusion pump oil, elongated a total of 3.3% at 1316°C (2400°F) under a stress of 15,000 psi for 133 hours after which the stress was increased to 16,500 and the test continued for an additional 24 hours. The same composition in the recrystallized condition, tested in ultra-high vacuum of 10^{-8} Torr at 1316°C (2400°F) and a stress level of 16,500 psi elongated 11.1% in 66.9 hours and had entered the third stage of creep 19 hours after application of the load.

TABLE 13 - Tensile Properties[a] of Non-Consumable Melted Ingots

Heat No.	Composition	Test Temperature °C	Test Temperature °F	Yield Strength 0.2% Offset (psi)	Ultimate Tensile Strength (psi)	Elongation, Percent Uniform	Elongation, Percent Total
NAS-6	8.2W-1.0Hf-0.035C	27	75	118,700	133,300	0.95	5.93
		27	75[b]	71,400	102,000	18.34	29.70
		1316	2400	37,500	55,200		23.00
		1650	3000	18,100	23,500		91.00
NAS-21	8.6W-0.53Hf-0.020C	27	75	125,600	138,200	0.95	4.75
		1316	2400	42,600	50,900		24.00
		1650	3000	17,200	20,500		86.00
NAS-16	11.3W-0.92Hf-0.06C	27	75[b]	101,800	126,200	14.68	22.30
NAS-38	7.1W-1.56Re-0.26Zr-0.02N	27	75[b]	106,600	118,500	15.65	25.90
NAS-39	7.1W-0.25Hf-1.56Re-0.13Zr -0.04N	27	75[b]	132,700	140,000	11.85	15.77
NAS-42	5.3W-1.56Re-0.65Mo-0.52Zr -0.08N	27	75[b]	141,000	151,400	13.72	17.58
NAS-36	5.7W-0.25Hf-1.56Re-0.7Mo -0.13Zr-0.017C-0.02N	27	75	160,200	168,700	0.84	2.87
		27	75[b]	127,100	134,000	13.03	23.85

Remarks: (a) Elevated temperature tensile tests performed by Metcut Assoc., Cincinnati, Ohio. See Appendix I.

(b) Annealed 1 hour at 1650°C prior to testing, all others tested in as-worked condition.

TABLE 14 - Summary of Creep Results for Tests at 1316°C (2400°F) and 10^{-8} Torr

Heat No.	Composition (w/o)	Condition	Stress (psi)	Test Time (hrs.)	Total Strain (%)	Minimum Creep Rate (%/hr.)
NAS-6	8.2W-1.0Hf -0.035C	Rx 1 hr. at 1650°C (3000°F)	14,770	172.0	1.35	0.00786
NAS-21	8.6W-0.53Hf -0.02C	Rx 1 hr. at 1650°C (3000°F)	15,070	96.1	1.22	0.0139
NAS-7	8.2W-1.0Hf -0.07C	Rx 1 hr. at 1650°C (3000°F)	14,530	96.4	1.57	0.0139
NAS-7	8.2W-1.0Hf -0.07C	As-worked (33% prior cold reduction)	14,974	72.2	11.40	0.08
NAS-8	8.2W-1.0Hf -0.1C	Rx 1 hr. at 1650°C (3000°F)	16,510	66.9	11.10	(b)
NAS-8(a)	8.2W-1.0Hf -0.1C	As-worked (33% prior cold reduction)	15,000	157.0	3.30	0.021

(a) Tested at 10^{-6} Torr in oil diffusion pumped system with liquid N_2 cold trap 133 hours of test at 15,000 psi at which time stress increased to 16,500 psi for balance of test.

(b) Started into third stage of creep after 19 hours.

D. TWO INCH DIAMETER INGOTS

The button study phase of the alloy development program which was described in the previous section culminates in the selection of the most promising button alloy compositions for melting as two inch diameter consumable electrode double vacuum arc melted ingots. Melting of the initial series of two inch diameter ingots was discussed in the previous quarterly report[3].

1. Chemical Analyses

Samples removed from the top and bottom portions of the conditioned ingots were analyzed for substitutional additions and interstitial additions and impurities. Results of the analyses are given in Table 15. During preparation of the first melt electrodes, the hafnium addition for NASV-2 was interchanged with that for the NASV-4 first melt electrode. Thus, the final compositions of these two ingots were slightly altered. The final composition for NASV-2 and 8W-2.0Hf-0.05C and for NASV-4, 8W-2.6Hf-0.37Zr and 0.05C.

Recovery of the metallic alloying additions was excellent. Carbon recovery was essentially 100%. No excess carbon was added to the electrode since the technique for making the carbon addition has been proven very effective.[11] The excellent homogeneity of the as-melted ingots is evident from the close agreement of the top and bottom analyses. Oxygen and nitrogen contents in the as-cast ingot were both low.

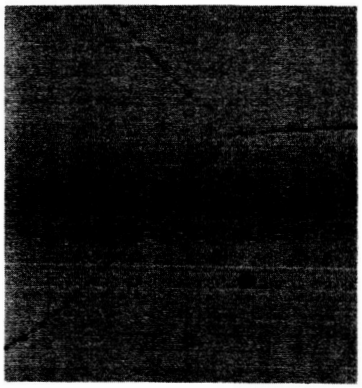
2. Metallography of As-Cast Ingots

Metallographic samples were taken from the top portion of each ingot. Representative as-cast microstructures are shown in Figure 15. The compositions which had the 0.05 a/o carbon addition (NASV-2, 4, and 5) exhibited similar as-cast microstructures, which consisted of grain boundary carbides plus a Widmanstätten type carbide precipitate. Increasing the carbon to 0.10 a/o (NASV-3 and -6) resulted in a continuous grain boundary carbide phase plus clusters of eutectic carbide.

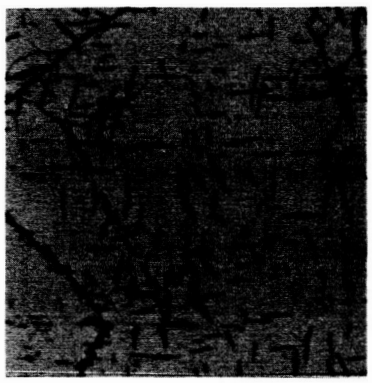
A series of one hour anneals at 1800-2400°C (3270-4350°F) were performed on as-cast samples to ascertain the changes in carbide morphology and also to estimate the approximate carbon solubility in the Ta-W-Hf matrix. The samples were helium quenched from the annealing temperature. All resulting microstructures retained the dispersed carbide phase indicating that the cooling rate from the annealing temperature was too slow to suppress precipitation during quenching or that the 2400°C (4350°F) annealing temperature was not high enough to allow complete solution of the carbon. The available Ta-C phase

TABLE 15 - Chemical Analysis of Two Inch Diameter Ingots

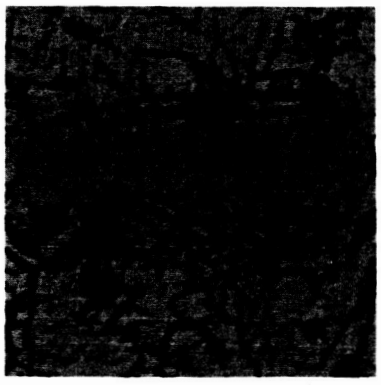
Heat No.	Nominal Composition (w/o)	Ingot Position	Analyzed, weight per cent					
			W	Hf	Zr	C	O	N
NASV-1	8W-2Hf (T-111)	Top	7.30	1.72	---	0.0016	0.0053	0.003
		Bottom	7.50	1.87	---	---	0.0050	0.002
NASV-2	8W-2.0Hf-0.05C	Top	7.70	1.74	---	0.054	---	0.002
		Bottom	7.75	1.83	---	0.051	---	0.002
NASV-3	8W-3.50Hf-0.10C	Top	7.60	3.35	---	0.098	0.0095	0.002
		Bottom	7.40	2.98	---	0.095	0.0089	0.003
NASV-4	8W-2.68Hf-0.37Zr -0.05C	Top	7.50	2.56	0.39	0.054	0.0038	0.003
		Bottom	7.30	2.59	0.38	0.048	0.0053	0.003
NASV-5	9.6W-3.15Hf -0.05C	Top	9.20	3.03	---	0.054	0.0032	0.003
		Bottom	8.60	2.69	---	0.049	0.0021	0.003
NASV-6	9.6W-3.90Hf -0.10C	Top	8.80	3.64	---	0.10	---	0.003
		Bottom	8.70	3.52	---	0.093	0.0059	0.003



(a) Ta-8W-2Hf
(NASV-1)



(b) Ta-9.6W-3.15Hf-0.05C
(NASV-5)



(c) Ta-8W-3.5Hf-0.10C
(NASV-3)

FIGURE 15 - Microstructure of As-Cast Ingots (Electrolytic Etch) 400X

diagrams[7,8] indicate that the alloys will be two phase at the highest annealing temperature investigated, 2400°C (4350°F).

After one hour at 1800°C (3270°F), the Widmanstätten precipitate in the compositions containing 0.05 a/o carbon tended to spheroidize. In the 0.1 a/o carbon compositions, the eutectic carbide was redistributed in a fine Widmanstätten type precipitate. Little or no change occurred in the morphology of the grain boundary carbides. After one hour at 2200°C (3990°F), the Widmanstätten precipitate in the 0.05 a/o carbon compositions had reprecipitated along sub-boundaries. A continuous sub-boundary carbide was formed in the 0.1 a/o carbon containing composition after one hour at 2200°C (3990°F). Again there did not appear to be any significant change in the morphology of the grain boundary carbide phase.

Although significant changes in carbide morphology were caused by the thermal treatments, annealing did not result in elimination of continuous carbide networks. Therefore, primary breakdown was scheduled to be accomplished on material in the as-cast condition.

3. Primary Working

Three-quarter inch thick slices from the top of each composition had been silicide coated and upset forged 50% on the Dynapak. The excellent upset forgeability of all six ingot compositions was described in the previous quarterly report.[3] The top and bottom faces of the upset slabs were lathe conditioned and the edges were ground.

Additional data on the upset forged slabs are contained in Table 16. Metallographic samples were taken from each slab. During abrasive cutting of NASV-3, a crack propagated in from the outer edge after the cut had been 50% complete. Further cutting on the slabs containing the 0.10 w/oC (NASV-3, and -6) was discontinued until after they had been annealed. After annealing, metallographic samples were taken from these two compositions without difficulty. The as-forged microstructure of NASV-1 (T-111 base line composition) contained a globular precipitate in the grain boundaries which is presumed to be HfO_2. The sheet produced from NASV-1 had a clean single phase microstructure and no evidence of a second phase was detected.

The remainder of the as-cast ingot was scheduled for extrusion to sheet bar using the high energy rate (Dynapak) extrusion. The as-machined billets were grit blasted and then a 0.005-0.010 inch thick coating of unalloyed molybdenum was applied to the billet by plasma spraying.

Heating of the coated billets to the extrusion temperature of 1800°C (3272°F) was by induction. An argon atmosphere was maintained around the

TABLE 16 - Upset Forged Ingot Top Data

Heat No.	Composition (w/o)	As-Forged Thickness (inches)	As-Conditioned Thickness (inches)	As-Forged Hardness DPH
NASV-1	Ta-8W-2Hf	0.455	0.392	---
NASV-2	Ta-8W-2Hf-0.05C	0.452	0.361	337
NASV-3	Ta-8W-3.15Hf-0.10C	0.466	0.397	---
NASV-4	Ta-8W-2.68Hf-0.37Zr -0.05C	0.458	0.377	361
NASV-5	Ta-9.6W-3.15Hf-0.05C	0.460	0.403	385
NASV-6	Ta-9.6W-3.90Hf-0.10C	0.459	0.358	---

billet during heating. Extrusion was through a zirconia coated sheet bar die. The total reduction by extrusion was nominally 4:1.

NASV-1, the T-111 base line composition extruded satisfactorily. However, the addition of carbon to the base composition apparently degraded the extrudability characteristics under the particular conditions used. During extrusion of NASV-5, premature firing of the ram resulted in an incomplete extrusion. The extrusion billet had not completely entered the container when the ram moved forward. As a consequence approximately two inches of extrusion was recovered and the balance of the billet was upset between the ram and anvil. Data pertinent to the extrusions are listed in Table 17.

Examination of samples taken from the failed extrusions indicated that poor extrudability may have been caused by the continuous grain boundary carbide phase. The microstructure of the as-extruded NASV-4 and NASV-6 was duplex, consisting of as-worked areas and areas where recrystallization had occurred. Since extrudability of the Ta-W-Hf matrix by high energy rate technique apparently was adversely affected by the carbon addition, primary breakdown by extrusion was discontinued. The remaining two ingots NASV-2 and NASV-3 were sectioned into thirds and upset forged. All pieces upset forged satisfactorily.

4. Secondary Working

The forged and annealed slabs were rolled to 0.06 inch thick sheet. Rolling results are in Table 18. Compositions NASV-1, -2, -4, and -5 were rolled at room temperature. NASV-3 and NASV-6 exhibited poor room temperature rolling characteristics. NASV-3 required warm rolling at 500°C (930°F). After 66.5% reduction, edge cracking initiated. Rolling was stopped, the sheet was conditioned, stress-relieved one hour at 1400°C (2550°F), and rolling to 0.06 inch thick sheet at 500°C (930°F) continued. NASV-6 was clad with an evacuated stainless steel can and initial breakdown rolling was done at 1200°C (2190°F). After a 60% reduction, the can was removed. Moderate to severe edge cracks had developed during initial breakdown. The 0.150 inch thick sheet was conditioned, stress-relieved one hour at 1400°C (2550°F) and rolling to 0.06 inches was done at 500°C (930°F). Examples of the as-rolled 0.06 inch thick sheet are shown in Figure 16.

The 0.06 inch sheet was annealed one hour at 1700°C (3090°F) and final rolling to 0.04 inches was done at room temperature.

5. Recrystallization

The one hour recrystallization temperature was determined on the 0.06 inch sheet rolled from upset forged ingot tops. The results of the one hour exposure at temperature on microstructure and room temperature hardness

TABLE 17 - Dynapak Extrusion Results

Heat No.	Composition	Billet Diameter (in.)	Billet Volume ($in.^3$)	Reduction Ratio	Fire Pressure (psi)	Energy x 10^{-6} in.-lbs.	Remarks
NASV-1	Ta-8W-2Hf (T-111)	1.773	5.3	4:1	800	1.32	Satisfactory extrusion
NASV-6	Ta-9.6W-3.90Hf -0.10C	1.763	5.23	3.8:1	800	1.32	Extrusion broke up into four pieces
NASV-4	Ta-8W-2.68Hf -0.37Zr-0.05C	1.771	8.62	4:1	1100	1.8	Extrusion broke up

Remarks:

Extrusion temperature - 1745°C (Optical-uncorrected)

Tooling - sheet bar die, zirconia coated

Lubrication - glass wool pad on die, aqua dag on liner wall

TABLE 18 - Results of Rolling Upset Forged Slabs to 0.06 Inch Thick Sheet

Heat No.	Composition	Starting Thickness (inches)	Initial Rolling Temp. (°C)	Final Rolling Temp. (°C)	Total % Reduction In Thickness	As-Rolled Hardness DPH
NASV-1	Ta-8W-2Hf (T-111)	0.392	R.T.	R.T.	84.7	337
NASV-2	Ta-8W-2Hf-0.05C	0.361	R.T.	R.T.	83.5	375
NASV-3	Ta-8W-3.5Hf-0.1C	0.397	500	500	85.0(a)	388
NASV-4	Ta-8W-2.68Hf-0.37Zr -0.05C	0.377	R.T.	R.T.	84.3	405
NASV-5	Ta-9.6W-3.15Hf-0.05C	0.403	R.T.	R.T.	85.3	423
NASV-6	Ta-9.6W-3.90Hf-0.10C	0.358	1200	500	83.5(b)	---

(a) Rolled to 0.130 inches, conditioned, stress-relieved one hour at 1400°C.

(b) Rolled to 0.150 inches, conditioned, stress-relieved one hour at 1400°C.

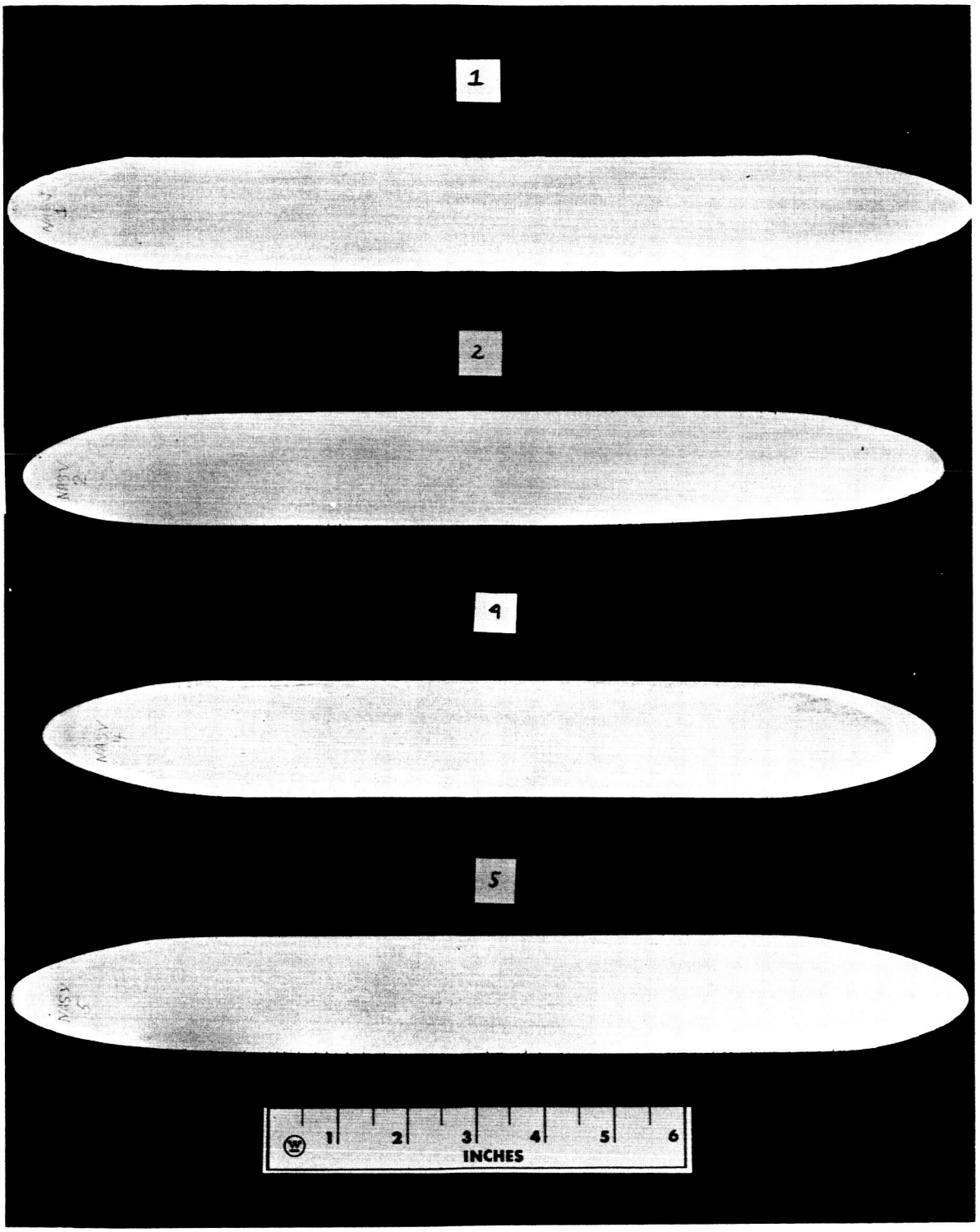


FIGURE 16 - As-Rolled Sheet Produced from Heats NASV-1,2,4, and 5

are listed in Table 19. Specimens were heated in vacuum ($<10^{-5}$ Torr) and quenched from the annealing temperature in helium. However, as was discussed previously, it was doubtful that the solvus was exceeded at the highest annealing temperature used and thus the carbon containing compositions were two phase at the annealing temperature. Both the addition of carbon and the addition of W + Hf to the base line T-111 (NASV-1) compositions caused an increase in the recrystallization temperature of approximately 100-200°C (180-360°F).

6. Weldability

TIG and EB welded specimens were prepared from as-rolled 0.04 inch thick sheet. Specimens were prepared and tested in bending to determine the effects of welding on the ductile-brittle transition temperature. Bend test results are shown in Table 20. The base line T-111 composition (NASV-1) was ductile at liquid nitrogen temperature over the nominal 2t bend radius. The addition of 500 ppm carbon to the T-111 composition (NASV-2) raised the temperature for a full 90° bend of the TIG welds to +500°F, and to room temperature for the EB welded material. However, both NASV-2 and -4 exhibited close to 90° bends at room temperature over 1.8t before cracking, and the cracks were confined to the weld metal area. The degradation of the as-welded bend ductility with respect to T-111 can be ascribed to the metallurgical changes observed in the weld metal and heat affected zone. The heat affected zone of NASV-2, -4, and -5 were single phase. Photomicrographs of the fusion zone, heat affected zone, and base metal are shown in Figure 17. This same behavior was noted and discussed on similar compositions welded under the button study phase. The extremely high hardness of this single phase heat affected and fusion zone is evident in the hardness traverses shown in Figure 18. The high hardness values are evidently a result of a solution of carbon in the matrix during welding and its retention in the lattice during cooling.

As-welded NASV-5 was aged for one hour at 1300°C (2370°F) and 1400°C (2550°F). However, both failed in bending at room temperature over a 2t bend radius. There was some improvement in ductility as in the as-welded condition, fracture occurred after bending through an angle of 10-15° while the aged specimens were bent approximately 50-70° before failure occurred. Aging studies are in progress to determine whether or not post-weld thermal treatments will significantly change the ductile-brittle temperature of the welded specimens.

The room temperature minimum bend radius was determined on the TIG welded specimens and is 3.5t for NASV-2 and 4t for NASV-4. NASV-5 was brittle over 4t at room temperature and failure occurred after bending through an angle of 16°.

TABLE 19 - Summary of Microstructure and Room Temperature Hardness After One Hour Anneals

Heat No.	Composition	As-Rolled(a)	Annealing Temperature, °C / °F						
			1200 / 2200	1400 / 2550	1500 / 2730	1600 / 2910	1700 / 3090	1800 / 3270	2000 / 3630
NASV-1	8W-2Hf (T-111)	337 W	283 RB	213 R	211 R	214 R	206 R	206 R	200 R
NASV-2	8W-2Hf-0.05C	375 W	334 W	262 R	276 R	271 R	269 R	266 R	278 R
NASV-3	8W-3.50Hf-0.10C	388 W	332 W	296 W	---	295 R	---	302 R	308 R
NASV-4	8W-2.68Hf-0.37Zr-0.05C	405 W	345 W	268 R	296 R	307 R	292 R	290 R	297 R
NASV-5	9.6W-3.15Hf-0.05C	423 W	362 W	281 RP	292 R	322 R	---	316 R	307 R

(a) Rolled 85% prior to annealing.

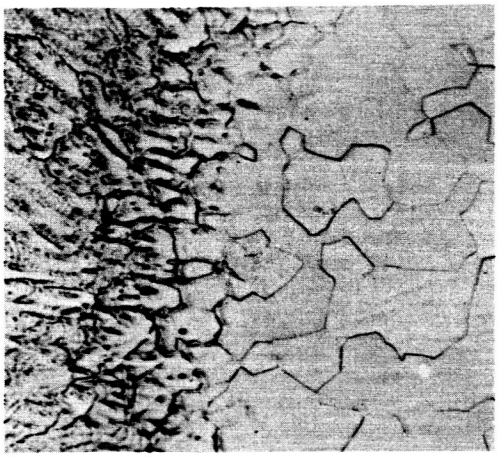
W - Wrought
RB - Formation of equiaxed grains $<$50% complete
RP - Formation of equiaxed grains $>$50% complete
R - Formation of equiaxed grains $>$95% complete

TABLE 20 - TIG Weld Bend Test Results

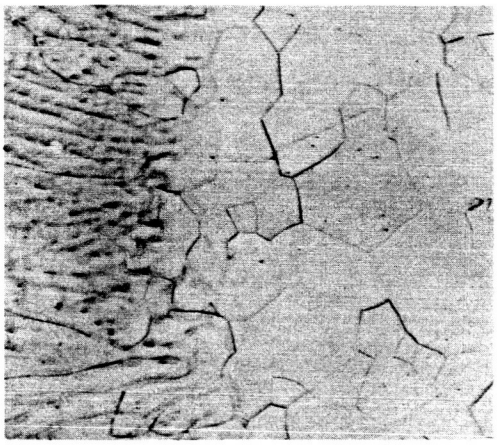
Composition (Heat No.)	Test Temperature (°F)	Bend Factor xt	Unloaded Bend Angle (°)	Remarks
Ta-8W-2Hf	R.T.	1.8	92	Ductile
(NASV-1)	-320	1.8	98	Ductile
Ta-8W-2Hf-0.05C	R.T.	1.8	91	Crack in weld metal
(NASV-2)	R.T.	4.0	89	Ductile
	R.T.	3.5	90	Ductile
	+200	1.8	86	Crack in weld metal
	+400	1.8	92	Crack in weld metal
	+500	1.8	91	Ductile
	-200	4.0	25	Crack in weld metal
Ta-8W-2.68Hf	R.T.	1.8	90	Crack in weld metal
-0.37Zr-0.05C	R.T.	3.5	79	Crack in weld metal
(NASV-4)	R.T.	4.0	94	Ductile
	+200	1.8	70	Crack in weld metal
	+400	1.8	92	Crack in weld metal
	+500	1.8	90	Crack in weld metal
Ta-9.6W-3.15Hf	R.T.	1.8	46	Crack in weld metal
-0.05C	R.T.	3.5	16	Crack in weld metal
(NASV-5)	R.T.	4.0	16	Crack in weld metal
	+200	1.8	10	Crack in weld metal
	+400	1.8	10	Crack in weld metal
	+500	1.8	20	Crack in weld metal

Remarks:

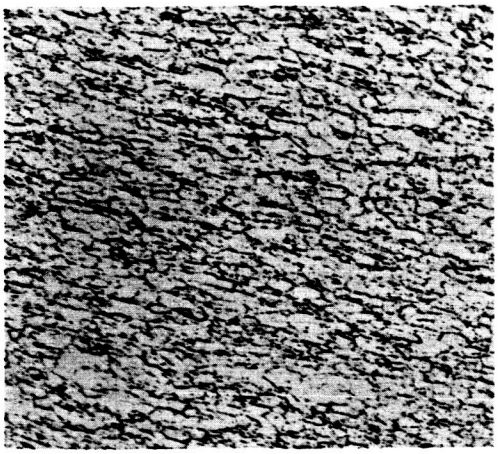
Specimen size - 12t wide x 24t long x 0.04 inches thick
Span width - 15t (0.6 inches)
Punch radius - 0.071 inches
Bend radius - ~2t
Ram speed - 1 inch per minute
Welding atmosphere - Argon with less than 5 ppm O_2



(a) Fusion and Heat Affected Zone, NASV-5



(b) Fusion and Heat Affected Zone, NASV-2



(c) Base Metal, NASV-2

FIGURE 17 - Microstructures of NASV-5 (Ta-9.6W-3.15Hf-0.05C) and NASV-2 (Ta-8W-2Hf-0.05C) (Electrolytic Etch) 150X

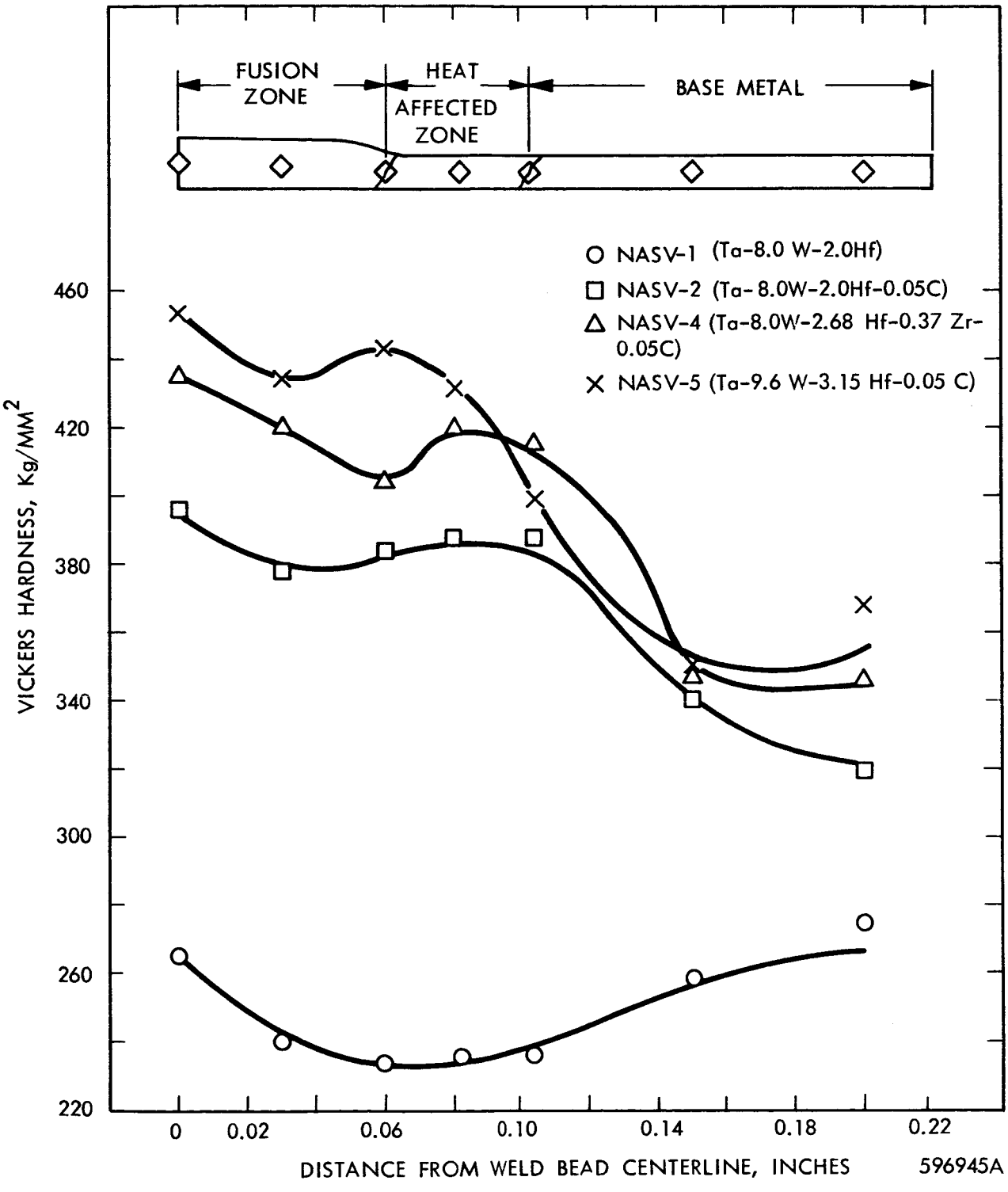


FIGURE 18 - Hardness Traverse of As-Welded Samples.

7. Base Metal Mechanical Properties

The room temperature tensile properties of the 0.04 inch thick sheet in the as-worked condition and after annealing for one hour at 1650°C (3000°F) are summarized in Table 21. Increasing the carbon and/or tungsten and hafnium content of the T-111 composition produced significant increases in strength with little decrease in tensile elongation. The room temperature properties of NASV-1 are comparable to the room temperature properties of T-111 as reported by Ammon and Begley.[9]

Fabricability also was not impaired by the carbon additions as the bend transition temperature of the base metal, after annealing for one hour at 1650°C (3000°F), was below liquid nitrogen temperature. This behavior was exhibited by material tested with the axis of rolling parallel and perpendicular to the bend axis. At room temperature the annealed sheet could be bent through an angle of 180° over 0-.5t without failure, with the rolling direction parallel or perpendicular to the bend axis.

Short time stress-rupture properties were determined on NASV-3, and NASV-5 to determine if the carbon addition which increased low temperature properties was an effective strengthener at elevated temperature. The stress-rupture data are in Table 22. Stress-rupture of NASV-3 and NASV-5 at 1316°C (2400°F) after annealing for one hour at 1650°C (3000°F) was essentially the same as reported for T-222 (Ta-9.6W-2.4Hf-0.01C).[9] However, annealing NASV-3 for one hour at 2200°C (3990°F) increased the stress-rupture life at 1316°C (2400°F) and 40,000 psi by a factor of 3. However, it should be emphasized that the long time creep behavior is the critical property under study and that large increases in short time properties, i.e. 10 hours, will not necessarily result in a like improvement of creep properties since the deformation mechanisms are considerably different.

Creep property determinations under ultra-high vacuum conditions have been initiated. NASV-1, the T-111 base line composition, exhibited a total of 2.63% strain in 73 hours at 1316°C (2400°F) under an applied stress of 14,010 psi. The hundred hour creep properties of the sheet from the two inch diameter ingot compositions are being obtained at three stress levels.

TABLE 21 - Tensile Properties of 0.04 Inch Sheet From Two Inch Diameter Ingots

Heat No.	Composition	Test Temperature		Yield Strength 0.2% Offset (psi)	Ultimate Tensile Strength (psi)	% Elongation	
		°C	°F			Uniform	Total
NASV-1	Ta-8W-2Hf	27	75	114,500	119,800	0.7	7.00
		27	75(a)	69,000	85,900	20.20	36.28
NASV-2	Ta-8W-2Hf -0.05C	27	75	136,400	147,250	0.88	6.55
		27	75(a)	74,600	105,000	15.65	21.75
NASV-4	Ta-8W-2.68Hf -0.37Zr-0.05C	27	75(a)	86,400	115,600	18.94	28.58
NASV-5	Ta-9.6W-3.15Hf -0.05C	27	75	155,700	171,200	0.83	5.58
		27	75(a)	107,900	129,500	18.57	28.67
NASV-6	Ta-9.6W-3.90Hf -0.10C	27	75	172,700	185,300	0.88	4.57
		27	75(a)	113,800	129,900	18.38	25.80

Remarks: (a) Annealed one hour at 1650°C prior to testing, all others tested in as-worked (33% prior reduction) condition. See Appendix I.

TABLE 22 - Stress-Rupture Results[a]

Composition	Test Temp. (°C/°F)	Stress (psi)	Rupture Time (hrs.)	Rupture Elongation (%)	Condition
8W-2Hf-(T-111)[b]	1316/2400	33,500	1.0	30.0	Rx 1 Hr. at 1650°C (3000°F)
8W-3.50Hf-0.1C	1316/2400	45,000	0.7	53.3	Rx 1 Hr. at 1650°C (3000°F)
9.6W-3.15Hf-0.05C	1316/2400	40,000	4.0	55.1	Rx 1 Hr. at 1650°C (3000°F)
8W-3.50Hf-0.1C	1316/2400	40,000	13.3	5.0	Rx 1 Hr. at 2200°C (3990°F)

Remarks:

(a) Tests conducted at 10^{-6} Torr

(b) T-111 data reported by Ammon and Begley[11]

III. FUTURE WORK

It is anticipated that all melting and fabrication to sheet stock will be completed during the next quarterly period. An additional five to six compositions will be selected based on data obtained from the 800 gram non-consumably melted ingots and will be melted as two inch diameter ingots and processed to 0.04 inch thick sheet. Heat treatment, hot hardness, and weldability studies will be largely completed for all the alloys. Creep testing will continue at a high level of effort.

IV. REFERENCES

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8. E. K. Storms, "A Critical Review of Refractories, Part I Selected Properties of Group 4-a, 5-a, and 6-a Carbides", LAMS-2674, March 15, 1962.

9. R. L. Ammon and R. T. Begley, "Pilot Production and Evaluation of Tantalum Alloy Sheet", 7th Quarterly Report under Contract N600(19)-59762, WANL-PR-M-008, Astronuclear Laboratory, Westinghouse Electric Corporation.

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11. R. L. Ammon and R. T. Begley, "Pilot Production and Evaluation of Tantalum Alloy Sheet", Summary Phase Report prepared under Contract NOw-62-0656-d, WANL-PR-M-004, June 15, 1963.

APPENDIX I

TEST SPECIFICATION

1. Tensile Tests, Elevated Temperature (2000-3500°F)

   a. Temperature measurement and verification, and strain rate shall conform to the requirements of MAB Report 192M, Paragraphs 1.7.2, 1.9.1, 1.9.2, and 1.9.3.

   b. In addition to the requirement of 1a, the following conditions will be met for the elevated temperature tensile tests:

      1. Pressure in the test chamber will not exceed 1×10^{-5} Torr during the test.

      2. The cold leak rate of the test chamber shall not exceed a pressure rise rate of 0.3 microns/minute.

      3. A liquid nitrogen cooled trap shall be used between the diffusion pump and test chamber to retard backstreaming of diffusion pump oil.

      4. Pre-test procedure will include the following:

         a. Dimensional measurements

         b. Specimen cleaning

            1. Degrease with acetone or other suitable solvent.

            2. Scrub with soap and/or alkaline cleaner.

            3. Hot distilled water, rinse.

            4. Acid dip - 5% HNO_3 - 2% Hf, balance water, R.T.

            5. Hot distilled water, rinse.

            6. Ethyl alcohol, rinse.

            7. Dry.

         c. Wrap (tight) gage length of test specimen with Ta foil.

         d. Attach thermocouple(s) to wrapped gage length.

e. Protect thermocouple hot junction from direct radiant heating from furnace with additional layer of loosely wrapped Ta foil.

5. Elevated tensile test specimen per Figure I-1.

2. Room Temperature Tensile Tests

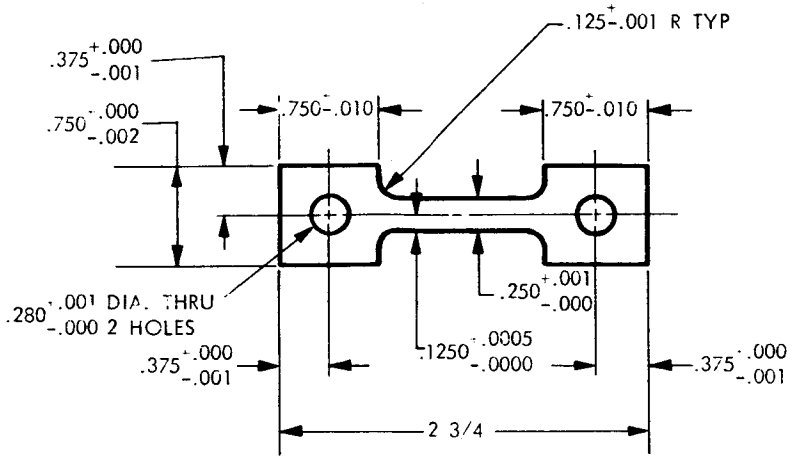
a. Requirements of (1) to apply where applicable except strain rate shall be 0.005 inches/inch through 0.6% offset yield strength and 0.05 inches/inch until fracture occurs.

b. Test specimen configuration per Figure I-2.

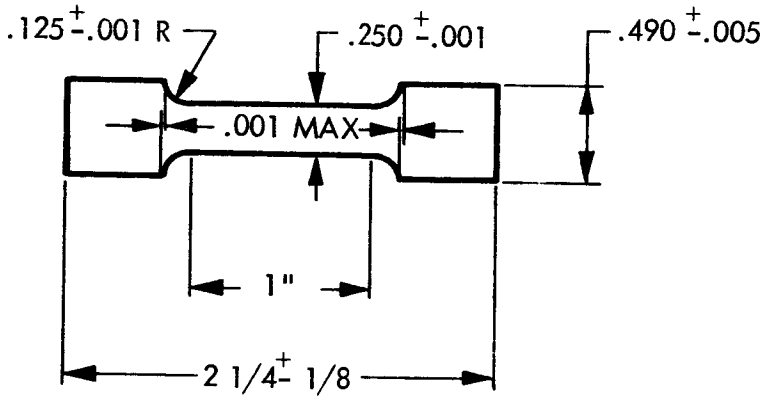
3. Creep Tests

a. Requirements of (1) to apply where applicable except pressure of test chamber shall be maintained at 10^{-8} Torr.

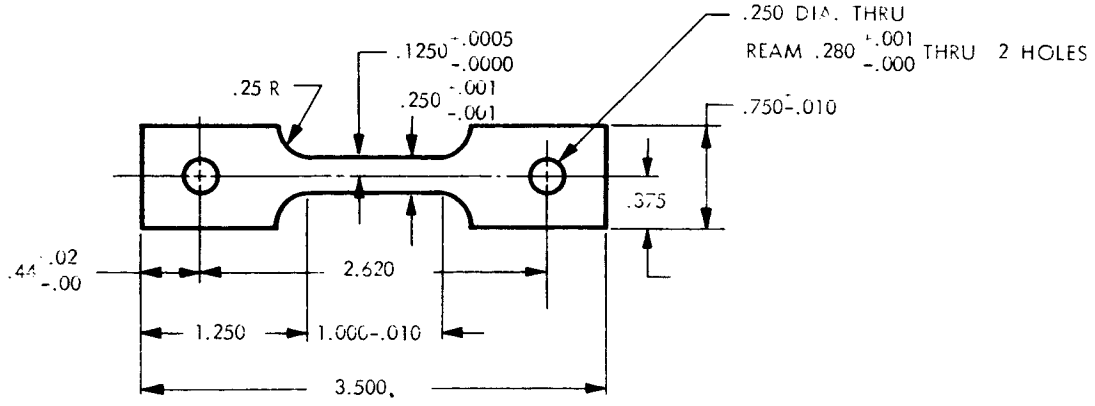
b. Test specimen configuration per Figure I-3.



1. Elevated Temperature Tensile Test



2. Room Temperature Tensile Test



3. Elevated Temperature Creep Test

FIGURE I - Test Specimen Configurations